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THE METALLURGICAL STRUCTURE AND MECHANICAL PROPERTIES AT LOW TEMPERATURE OF NITRONIC 40, WITH PARTICULAR REFERENCE TO ITS USE IN THE CONSTRUCTION OF MODELS FOR CRYOGENIC WIND TUNNELS

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The Metallurgical Structure and Mechanical Properties at Low Temperatures of Nitronic 40, with Particular Reference to its Use in the Construction of Models for Cryogenic Wind Tunnels

1. INTRODUCTION

Nitronic 40 was one of the materials reported on in an excellent report prepared by Tobler of the Fracture and Deformation Division of the National Bureau of Standards, "Materials for Cryogenic Wind Tunnel Testing" (Ref.1). Subsequently, at a conference held at NASA Langley Research Center (LaRC)(Ref.2) in November 1979, Hudson (Ref.3) presented a more detailed analysis of the criteria applied for selecting materials for the construction of Pathfinder I, the first model scheduled for use in the National Transonic Facility (NTF) presently under construction at LaRC. Pathfinder I is a research and development model intended to show up problem areas associated with testing models at high loads and cryogenic temperatures in order to achieve full scale Reynolds No. test condition. Hudson's primary criteria included strength, toughness and availability in the required product forms, while secondary considerations covered corrosion resistance, machinability, cost and delivery.

As a result of the analysis by Hudson, Nitronic 40 was judged to be the only acceptable material available for fabricating Pathfinder I and the requisite material was duly ordered. This lead was followed by some U.S. aircraft manufacturers interested in producing models for evaluation in the NTF, and Nitronic 40 plate is known to have been delivered to McDonnell Douglas and Lockheed Georgia.

All available information emphasised the high austenite stability of Nitronic 40, even after severe cold working and cryogenic cycling. Furthermore excellent toughness at low temperatures appeared to be indicated by a Charpy V notch impact energy in excess of 65ft-lbs at -320F (-196C). However, Charpy tests carried out on samples cut from the 5.5 inch thick plate obtained for the wing of Pathfinder I, and intended to check the heat-treatment used at Langley to relieve machining stresses, showed surprisingly low impact energies in the range 20-25ft-lb at -275F (-170C). As these values were relatively close to the 25ft-lb acceptance criterion set for NTF models no undue alarm was felt and the low readings were attributed to the large grain size exhibited by this plate.

The potentially more serious nature of the problem first became apparent when a LaRC technician examining metallographic sections taken from pins nitrided to prevent galling, noticed that the grain boundaries contained precipitates and second phase particles that suggested that the material was in a sensitised condition. Shortly afterwards a more detailed metallurgical analysis of the Nitronic 40 used at Langley was initiated to help evaluate warpage problem that had occurred with 15-5PH stainless steel models tested in the 0.3m Transonic Cryogenic Tunnel (TCT). This revealed that much of the Nitronic 40 currently being fabricated into parts of Pathfinder I was indeed severely sensitized and, furthermore, that it also contained substantial amounts of delta ferrite.

The presence of ferrite in a material that is supposed to be fully austenitic places severe limitations on its suitability for use in cryogenic applications, and hence a comprehensive study has been carried out to determine the extent of the problem. This report discusses in detail the results of these investigations and their implications for the future use of Nitronic 40 in Pathfinder I and other models intended for use in the NTF and other cryogenic wind tunnels.

2. NITRONIC 40, A NITROGEN-STRENGTHENED, HIGH-MANGANESE, AUSTENITIC STAINLESS STEEL.

The 300 series austenitic stainless steels containing at least 18% chromium and 8% nickel have long been used successfully for cryogenic applications, but one of their major limitations has, however, been their relatively low yield strengths at room temperature. This has been improved in the case of grades 304N, 316N and 347N by the addition of 0.2% nitrogen, and Nitronic 40 may be considered to be a further development in this direction. It also contains a slightly higher chromium content of 21% to improve corrosion resistance and a lower nickel content of 6% but, despite this reduction in nickel, austenite stability is ensured by the very much higher manganese level of 9%. These major alloying compositions are reflected in its original ARMCO designation, 21-6-9 and a full chemical specification is given in Table 1, together with the compositions of the materials used in this investigation.

Thanks largely to the high nitrogen content, the room temperature yield strength of Nitronic 40 is nearly twice that of types 304, 321 and 347 and it also has good elevated temperature properties. More relevant to this study is its excellent nominal mechanical properties at cryogenic temperatures, as illustrated by the data in Table 2 which was taken from ARMCO Bulletin No.S-54a. Despite its higher strength, it can be formed using similar techniques to those used for 300 series stainless steels and it can be welded by conventional welding methods using appropriate fillers. Furthermore, it is readily rolled or forged into a variety of product forms and even after severe cold working it has an extremely low magnetic permeability - an indication of its high austenite stability.

The only apparent limitations noted by ARMCO in Product Data Bulletin No.S-54a (Ref.4) are the formation of carbides and sigma phase during prolonged holding at temperatures in the range 1000-1600F (540 to 870C), detailed schedules being given in Tables 3 and 4 for carbide and sigma formation respectively. Such limitations are also found for most 300 series stainless steels and their avoidance should present no serious design problems when used in conventional applications. As we shall see, they do, however, pose more serious problems when heat treatments are required to remove machining stresses in high precision components.

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Nitronic 40 appears therefore to be an eminently suitable choice of material for cryogenic wind tunnel applications such as the Pathfinder I model. It is nevertheless a relatively new alloy without a long history of use in high load-bearing cryogenic applications that require excellent dimensional stability, and it is reasonable to expect that careful quality control and materials testing procedures should be carried out in order to detect any difficulties at the earliest possible stage.

The occurrence of small, but highly damaging, percentages of delta ferrite in a supposedly fully austenitic material is, however, not so lightly dismissed. Nowhere in the technical literature supplied by the manufacturer, or otherwise readily available, is the possibility of the existence of this second phase even mentioned. An expert in the welding metallurgy of stainless steels might expect to find delta ferrite in the weld or heat-affected-zones as its presence is often encouraged to combat the problem of hot cracking (slivering), but as-received plate or bar should contain only the fully stabilised austenite phase.

As the results detailed later in this report show, once present it is almost impossible to remove delta ferrite without creating metallurgical problems of even greater severity. Material already bought and models either partially or wholly constructed are therefore unlikely to be capable of use without delta ferrite in their structure, and this will have to be taken into account when determining their operating limits.

Furthermore, if Nitronic 40 is to be used widely for applications at cryogenic temperatures, procedures will have to be devised to ensure that it can be supplied in a completely ferrite-free condition.

3. CARBIDE AND SIGMA PHASE FORMATION IN AUSTENITIC STAINLESS STEELS

3.1 Carbide precipitation;sensitization

As may be seen from Figure 1, a pseudo-binary phase diagram for the (Fe,Cr,Ni)/MC system, the solubility limit for carbon increases slowly with temperature from 0.02% at 930F(600C) to 0.05% at 1470F(800C) and then much more rapidly as the temperature rises. As austenitic stainless steels rarely have carbon contents in excess of 0.1%, a solution heat-treatment at temperatures in the range 1920-2100F(1050-1150C) is sufficient to allow all the carbon to be taken into solution.

The structures formed on subsequent cooling depend on the kinetics of a nucleation and diffusion-controlled growth mechanisms that are conveniently described by a Temperature,Time, Transformation (T.T.T) curve of the form shown in Figure 2. Thus, if the solutionized material is quenched from say 1150F rapidly enough to avoid intersecting either of the two carbide curves, the carbon will remain locked in a supersaturated solid solution evenly distributed interstitially throughout the steel. If, however, the cooling rate is much slower, or if the quenched material is reheated and held for any appreciable length of time at temperatures in the range 1000-1600F (550-850C), carbides will be formed which nucleate preferentially at the austenite grain boundaries. In the 18Cr-8Ni and 21Cr-9Mn-6Ni stainless steels, these carbides are predominantly of the $(FeCr)_{23}C_6$ type and the chromium is obtained by depleting a zone adjacent to the grain boundary as indicated schematically in Figure 3. Such depletion has been measured by electron microprobe analysis and in Figure 3 a typical chromium level trace is shown to decrease from about 18% at the centre of a grain down to a minimum value of less than 12% in the depleted zone before peaking in the $(FeCr)_{23}C_6$ particle and then repeating this pattern on the other side of the grain boundary.

This behaviour has two important consequences for the properties of the material: one chemical, the other mechanical.

3.1.1 Corrosion resistance

Stainless steels require a minimum of 12% chromium to give them the corrosion resistant properties suggested by their name. In the depleted zones adjacent to the grain boundaries, the chromium level drops below 12% and so these depleted zones are liable to chemical attack in the form of intergranular corrosion. This problem is widely known as "weld decay" as it is most prevalent in the heat-affected-zones found approximately 0.25" (6mm) on either side of welds that have been exposed to temperatures in the range 1100-1600F (550-850C). Subsequent exposure to a mildly corrosive environment, sea water for example, allows the depleted zones to be eaten away to leave the grains isolated from each other. (The classical analogy is to liken the effect to that of degrading the strength of the mortar in a brick wall so that the bricks are only held together by compacted sand). The fracture surface shown in the Scanning Electron Microscope view of Figure 4 illustrates dramatically the intergranular nature of failure due to this corrosion mechanism.

A number of metallurgical solutions have been used to combat weld decay in 18/8 austenitic stainless steels. Lowering the carbon content to less than 0.03%, as in the 304L and 316L grades, reduces the amount of "free" carbon able to diffuse and join with chromium to form carbides and cause depleted zones. An alternative approach, which can be explained by reference to Figure 2, is to add voracious carbide forming elements, such as titanium (type 321) and niobium (type 347). The nose of the T.T.T. curve for Nb(Ti) carbides is at approximately 2100F (1100C) and heat-treatment at this temperature allows these carbides to form preferentially and combine with the carbon that would otherwise have produced the $Cr_{23}C_6$ carbides. Similarly, during continuous cooling from higher temperatures the Nb(Ti) carbides will form before the $Cr_{23}C_6$ types.

In the context of the 21-9-6 Nitronic 40 austenitic stainless steels, carbon contents are specified as a maximum of 0.04% and it may be seen from Table 1 that the highest value found in this investigation was 0.032%. As some authorities suggest that carbon levels <0.02% are required for complete freedom from carbide formation, titanium or niobium might profitably be added to Nitronic 40 to offer additional protection against the "weld decay" type of degradation to improve resistance to corrosion and sensitization.

3.1.2 Sensitization

Although the loss of corrosion resistance caused by chromium depletion is the most serious consequence of carbide precipitation for room temperature applications, it is the presence of the carbides in the grain boundaries that is of immensely greater significance for cryogenic applications. The carbides are responsible for a drastic loss of toughness because they embrittle the grain boundaries and lead to an intergranular mode of fracture instead of the more normal energy absorbing transgranular mode. This effect is known as "sensitization" and the characteristic appearance of the intergranular mode of failure in sensitized Nitronic 40 may be seen in Figure 5. In Figure 5a the Stereoscan view taken at x400 shows a ridge that corresponded to the intersection of two grain boundaries, while the higher magnification view of Figure 5b shows on each flank many small "ductile dimples", each of which probably nucleated on a carbide precipitate in the grain boundary. It is the ease with which the carbides act as fracture nuclei that causes the loss of toughness in sensitized stainless steels.

Before going into a more detailed consideration of sensitization in Nitronic 40, it is necessary to consider a second mechanism by which embrittlement may occur, namely sigma phase formation.

3.2 Sigma Phase Formation

Sigma phase is a hard (800VPN), brittle, non-magnetic, intermetallic compound FeCr which is also formed in Cr-Ni stainless steels by high temperature heat-treatment. The composition ranges over which it is stable at 1470F(800C) are shown in the ternary Fe-Cr-Ni diagram in Figure 7. In general, its formation is encouraged by increasing concentration of chromium and silicon and discouraged by increasing the content of nickel and other austenite stabilizers.

A further characteristic of particular relevance is the very much greater probability of sigma phase formation in duplex steels containing delta ferrite. Nucleation of sigma phase occurs preferentially at the austenite/ferrite grain boundaries and the ferrite, being richer in chromium, is absorbed during growth of the sigma phase. Furthermore, as there is a considerable decrease in volumes during this process, fine cracks tend to be formed in the delta ferrite.

Sigma phase is also nucleated at grain boundaries and the data in Table 4 indicates that it is formed over approximately the same temperature range, 1100-1600F(540-880C) as the grain boundary carbides, although the maximum sigma formation rate is at a slightly higher temperature than that for carbides. Hence in a component that is slowly cooled through this temperature range both carbide and sigma phase can be precipitated. It is rather difficult to distinguish them easily, especially at low concentrations, but they both cause embrittlement which increases in severity as their concentration rises.

A rather dramatic illustration of this effect is given by the Stereoscan views shown in Figure 6. This sample had been deliberately sensitized by heat-treatment for 168 hrs at 1380F(750C) and then subjected to a Charpy impact test at a temperature of -320F(-196C) in which it developed an impact energy of slightly less than 5ft-lbs! The highly intergranular nature of the fracture surface is readily apparent in the lower magnifications, while the multiple initiation sites may clearly be seen in the higher magnifications. Comparison with Figure 4 shows considerable similarity between the two fracture surfaces.

3.3 The occurrence of carbides and sigma phase in Nitronic 40 samples examined during this investigation

In order to give some standard by which the degree of sensitization present in the various Nitronic 40 samples may be judged, a series of specimens were subjected to heat-treatment at 1380F(750C) for increasing periods of time. Unfortunately due to the lack of material available, these heat-treatments were carried out on sample S, which had already undergone heat-treatment at Langley involving furnace cooling from 1950F(1065C). This procedure had already induced a degree of sensitization and hence the heat-treatments carried out at Southampton created additional carbide and sigma phase precipitation. The appearance of this ST series of specimens (as defined in Table 5) when polished and etched in mixed acids is illustrated at a magnification of x300 in Figures 8(a-e). It can be seen that after the LaRC Heat Treatment cycle the precipitates are in isolated groups in the grain boundaries and that with increase in holding time at 1380F(750C) their density increases until eventually they form an almost continuous network.

The degree to which various samples of Nitronic 40 studied in this investigation have been sensitized is illustrated in Figures 9(a-h). Figure 9a) represents Sample SH7 which had been desensitized by holding at 1950F(1065C) for 30 minutes and then quenching in liquid nitrogen. The grain boundaries are clear and completely free of precipitates. Figure 9b) is for Sample DA which is representative of the condition of the 60x44x5.5in thick slab as delivered to LaRC. Although this specimen has been somewhat over-etched to bring up the grain boundaries, the precipitates are clearly visible, and hence the material had a certain amount of sensitization before its heat-treatment at LaRC. Figure 9c) shows the condition of a Charpy test specimen SCVT4 on which sensitization in material that had been put through the LaRC-recommended heat-treatment to remove machining stresses was first identified by LaRC personnel and the author in July 1981.

Of possibly even greater significance is the amount of sensitization present in Specimen WA shown in Figure 9d), as this sample was taken from material adjacent to the leading edge of the left hand wing of Pathfinder I. As the thickness in this region had been reduced to about 2.5 inches by rough machining, it cooled more rapidly from the soaking temperature of 1950F(750C) and it had been hoped that this would avoid sensitization. Unfortunately, Figure 9d) shows this not to be the case as there is still a significant degree of precipitation in the grain boundaries.

The extent of the corporate misery is revealed by Figures 9e) and f) which are taken from 1.25in. thick Nitronic 40 plate supplied to McDonnell Douglas and Lockheed-Georgia respectively. In the case of the McDonnell Douglas material, the problem was identified during a visit to Long Beach by the author in August 1981, and a remedial heat-treatment was agreed upon and subsequently carried out in September 1981. This involved heat-treatment at 1950F(1064C) for 60-90 minutes, followed by quenching into liquid nitrogen of a plate 40in.long x 12in. wide x 1.25in.thick. According to a report received from McDonnell Douglas, distortion induced by the cryoquench was negligible and subsequent machining satisfactory. Incidentally, this is, to the best of our knowledge, the largest sample of Nitronic 40 that has been quenched from +1950F to -320F(+1065 to -196C). It would appear, therefore, that the Nitronic 40 model being produced by McDonnell Douglas should not be embrittled due to carbide or sigma-phase precipitation.

In contrast, it appears that the airfoil model fabricated for Lockheed-Georgia was competed before the sensitizing problem became fully apparent and that it would now be impractical to attempt any remedial metallurgical heat-treatments.

Finally, Figures 9g) and h) show at the higher magnification of x600 two views of the morphology of the grain boundary precipitates present in Sample SA after its LaRC heat treatment cycle. Figure 9g) illustrates a characteristic jagged linear feature visible in many of the sensitized samples, while in Figure 9h) precipitates are visible in the austenite grain boundaries and also at the interface between austenite and delta ferrite.

Nitronic 40 is still a relatively new alloy and little or no information is available on the detailed morphology of carbide or sigma phase precipitation. As noted earlier, the ST series of samples were sensitized by holding at 1380F(750C) for periods of 5, 24, 72 and 168 hours and Figures 10a)-h) show the changing nature of the precipitates with treatment time. Figure 10a) demonstrates the existence of discrete islands of sigma phase in the grain boundaries with semi-continuous carbides between them. After 24 hrs. over 50% of the grain boundaries contain almost continuous sigma and carbides, while the 72hr. specimen of Figure 10e) indicates pronounced development of the jagged linear feature noted earlier. Finally, after 168 hours the sigma phase precipitates in the grain boundaries have grown larger and more distinct as can be seen from Figure 10g). There seem to be indications that some grain boundaries contain more sigma phase while others have more carbides, but more work would be necessary to find out whether this effect was genuine or just coincidental.

The presence of discrete particles of sigma phase in the grain boundaries as shown in Figure 10g), allows us to interpret more fully the detailed appearance of the Charpy fracture surface of this specimen that was shown in Figure 6, particularly views g) at a magnification of x1.2K and h) at x3K. In 6g), the fine structure of the fracture surface has a 'rectangular' appearance compared, for example, with the more rounded 'ductile-dimple' nature shown for the partially sensitized specimens seen in Figures 5b) and 23h). One possible interpretation of this effect is that the fracture nuclei in the partially sensitized specimens are carbides, whereas in the heavily sensitized material they are the larger sigma phase precipitates. Once again, further work would be necessary to check the validity of this hypothesis.

As noted earlier, sigma phase formation is enhanced by the presence of delta ferrite and Figures 10b), d), f) and h) illustrate the much larger scale of the sigma phase formed within the delta ferrite as opposed to the grain boundaries. The higher chromium content of the delta ferrite, which is largely responsible for the enhanced rate of sigma formation, can be measured by electron microprobe analysis, and Table 6 gives the results found for a Charpy specimen, SHT4. Comparison with the macroanalysis for the same material reveals that there is indeed a movement of chromium into the delta ferrite from the surrounding austenite and that this is accompanied by the segregation of nickel in the opposite direction.

3.4 The effect of sensitizing and desensitizing heat-treatments on the Charpy impact energy of Nitronic 40 samples

While noting that Charpy impact test results are not by themselves adequate for determining the fracture toughness of a material, they do allow a useful and relatively inexpensive indication of the effect of different mechanical or thermal treatments on its toughness. Accordingly, Charpy tests have been carried out on many of the samples of Nitronic 40 investigated so far as part of the programme to develop models for cryogenic wind tunnels.

Knowledge of the location of the test bars and of the orientation of the bar axis and notch directions with respect to the rolling direction and plate thickness, is of critical importance in interpreting the large amount of data generated. Therefore in Figures 11, 12 and 13 we have attempted to define, to the best of our knowledge, these characteristics for the specimens whose results are considered in this report, while the results themselves are given in Tables 7 and 8.

It is convenient to start both logically and chronologically by considering the results for the SCV series of samples cut by LaRC from the 60x44x5.5in plate, billet number 69310-1F. As Figure 11 shows, these were cut from a triangular section from the top right hand corner, together with tensile test pieces.

> The Charpy bars are oriented with their long axis in the through-thickness direction and they are therefore conventionally described as SHORT TRANSVERSE or THROUGH-THICKNESS samples.

Two notch orientations are utilized and specimens with the notch cut parallel to rolling direction have been designated CVT1-12 and those with the notch cut at 90^{o} to the rolling direction CVL1-12. In any rolled plate, through-thickness specimens will exhibit lower toughness than either longitudinal or transverse samples because inclusions, second phases and any rolling defects will tend to be elongated along the rolling direction and the fracture initiated at the notch root will tend to propagate along these features. Furthermore, if the plate has not been extensively cross-rolled these features will tend to be elongated more extensively in the rolling direction than in the transverse direction and anisotropic mechanical properties will develop, hence the notches cut in different directions for specimens CVL and CVT. As the Nitronic 40 material under investigation contains substantial amounts of delta ferrite, its orientation and aspect ratio will play an important role in influencing the mechanical properties at liquid nitrogen temperatures. This feature will be considered further in Section 5.

Reference to Table 7 will show that the first twelve Charpy bars were tested at -275F(-170C) and that they represented three different heat-treatment cycles likely to be encountered during manufacture of Pathfinder I, namely combinations of stress-relieving heat-treatments at 1950F(1065C) and brazing cycles to 1850F(1010C). There is probably little or no significance between these different treatments with the majority of readings in the range 19-25ft.lb which indicate a severe degree of sensitization.

The remaining five specimens were cryocycled between room and liquid nitrogen temperatures before testing at -320F(-196C) to give impact energies ranging from 14 to 21ft.lbs. If these lower values were a result of the cryocycling, then there would indeed be cause for alarm as a model may experience such cryocycles many times during its operating lifetime. However, it is much more likely that the decrease is due to the lower testing temperature -320F(-196C), instead of -275F(170C) and if so the series of readings taken at -275F should be ratioed downwards by a facto about 0.75. (This factor is the ratio of the average impact energy of the five specimens tested at -320F(16.8ft.lb) to the average energy of the twelve samples tested at -275F(22.6ft.lb). Support for this interpretation comes from metallographic and fractographic analysis of representative samples from the two sets of tests which failed to show any factors which could be attributable to the effect of cryocycling, e.g. there was no evidence of pre-cracking in the delta ferrite, grain boundary crack nucleation or partial transformation to the more brittle martensitic phase. Furthermore, the amount and orientation of the delta ferrite present in the plane of fracture beneath the notch root would also increase the spread of values obtained from any series of tests and therefore mask effects due to differing heat-treatment cycles.

If we assume for the purpose of further analysis that the average impact energy of all 17 samples from the SCV series would have been about 16-17ft-lbs if tested at -320F, then these values can be compared with the average of 30ft.lbs given in Table 8 for the three specimens SHT4, 5 and 6. These specimens were given a desensitizing heat-treatment at Southampton consisting of a

?0-minute soak at 1950F(1050C), followed by quenching into liquid nitrogen to ensure a rapid cool through the sensitizing temperature range of 1700-1100F(920-590C). Comparison is possible between these two sets of tests as both utilized Charpy bars with their long axes oriented in the through-thickness direction of the 5.5in plate. It is therefore an indication of the degree of embrittlement caused by the Langley heat-treatment cycles which specify furnace cooling after soaking rather than the liquid nitrogen quench used at Southampton or the rapid air cooling and water or oil quenching for heavier sections specified by the manufacturers, ARMCO. This point will be reconsidered in more detail later in this report.

Further valid comparison can be made between the three specimens SHL1, 2 and 3 also desensitized at Southampton, which had an average impact energy of 84ft.lb at -320F, and the four samples T3 LS, LT, TS and TT. These were taken from the #3 dimensional stability test, 6in.x8in.x0.625in. airfoil and gave an average impact energy of 78.5ft.lb. at -320F. At this stage, the origin of the Nitronic 40 used for this airfoil is uncertain but it is reasonable to assume that the airfoil's axis of symmetry lay in the plane of the plate, probably orientated along the major rolling axis. Thus these two sets of results are believed to be representative of the toughness of desensitized Nitronic 40 plate in the longitudinal and/or transverse orientations.

Another important series of results are those obtained from an offcut of the 1.25in thick plate supplied to Lockheed-Georgia. As the plate thickness was insufficient to allow through-thickness Charpy bars to be machined, only longitudinal (L) and transverse (T) oriented specimens were taken. Notches were cut either through-thickness (-T) or in the plane of the plate (-S) in order to investigate the degree of anisotropy in toughness (see Figure 13). This material is thought to have undergone the same heat-treatment as the LaRC specimens, i.e. furnace-cooled after soaking, and the sensitization therefore to be expected is in fact confirmed metallographically as shown in Figure 9f). As will become apparent in Section 5, the delta ferrite in this material is more heavily orientated in the 1.25in thick plate than in the 5.5in plate and so direct comparisons between the results from the Lockheed material and those described in the previous paragraph are not strictly possible. Nevertheless, if anything the thinner plate should be tougher and thus the average impact energy of the fifteen sensitized Lockheed samples (37.2ft.lb) should be compared with a probable desensitized value in excess of the Southampton average of 84ft.lb and the LaRC average of 78.5ft.lb. This prediction could be easily checked if further Charpy bars were cut from any remaining offcuts from the Lockheed Nitronic 40 after they had been put through a desensitizing cycle of soaking at 1950F(1060C) followed by quenching into liquid nitrogen.

> The conclusions to be drawn from this series of tests is that sensitization has lowered the Charpy impact energy at -320F by approximately 50%, and that this condition has been brought about by cooling slowly

through the critical temperature range after plate fabrication and after the LaRc stress-relieving heat-treatment at 1950F.

Further indications of the degree of sensitization induced by the LaRC heat-treatment cycle can be obtained from the results of tests on the sigma heat-treated (ST) series of specimens which are also given in Table 8. Heat treatment times of 5, 24, 72 and 168 hours gave average impact energies of 22.5, 12.2, 7.0 and 4.5ft.lb respectively, a dramatic indication of just how severely embrittled a normally tough material like Nitronic 40 can become if maltreated!

As all the Charpy specimens for the ST series of tests were cut from the same material and in the same orientation as the SA specimens which had undergone the LaRC Stress Relieving cycle, and the SHL specimens that had been desensitized, it is possible to judge the degree of sensitization caused by the LaRC SR cycle. Thus:

Treatment	Average Impact Energy
Desensitizing heat treatment	84 ft.lbs.
Langley SR " "	45 " "
SR + 5hr at 1380F	22.5 " "
SR + 24hr at 1380F	12.2 " "
SR + 72hr at 1380F	7.0 " "
SR + 168hr at 1380F	4.5 " "

These experimental values give an excellent fit to the relationship

$$CV \propto A(SR + ST)^{-N}$$

where CV is the Charpy impact energy at -320F in ft.lb.
A is a constant.
SR is an equivalent time at 1380F(750C) for the LaRC HT cycle.
ST is the heat-treatment time at 1380F(750C)
N is a dimensionless constant.

Figure 14 shows these results plotted graphically using log-log scales and assuming values of 1, 2 and 3 hours for SR. It can be seen that a very good fit is obtained for the assumed value of SR=2, which then gives A as 60ft.lb and N=-0.5.

It is in fact possible to rationalize the value of -0.5 for the exponent N, as the rate controlling factor in the formation of carbides and sigma phase in the grain boundaries will be the diffusion of carbon, chromium, nickel and other segregating elements. At a constant temperature the basic relationship given by Ficks law will be,

$$\text{Number of precipitates} \propto (Dt)^{0.5}$$

where D is the diffusion constant at the reaction temperature and t is the reaction time.

Assuming that the toughness as measured by the Charpy impact energy is inversely proportional to the number of precipitates gives,

$$\begin{aligned} CV &\propto 1/\text{No. of precipitates} \\ &\propto 1/(Dt)^{0.5} \end{aligned}$$

i.e. Charpy energy $\propto$ constant x time$^{-0.5}$.

More extensive experimental work would of course be necessary to check the validity or otherwise of this argument!

3.5 Possible Heat-treatment Cycles for Nitronic 40 to Desensitize, Stabilize and Relieve Machining Stresses

3.5.1 Cryoquenching using liquid nitrogen

So far in Section 3 we have seen the deleterious effect of carbide and sigma phase precipitation on the mechanical properties of Nitronic 40 at low temperatures. Thus, as most material studied in this investigation was at least partially sensitized on delivery, it is necessary to specify a heat-treatment cycle capable of allowing the material to develop the properties indicated by the manufacturers literature. Furthermore, experience at LaRC has shown that heat-treatment is necessary in order to put the material into a "deadened" condition, that is to remove any residual stresses that would otherwise cause distortion when layers of metal are removed during rough machining. Finally, it was also anticipated that further stress-relieving treatments might be necessary to remove any additional stresses that are introduced during rough or intermediate machining.

There is, however, a conflict of requirements which has contributed to the problems which have beset the Pathfinder I and other models for operation in cryogenic wind tunnels. On one hand, rapid cooling through the critical temperature range is required in order to minimize the possibility of carbide or sigma phase formation, while on the other hand slow cooling rates are normally recommended to minimize quench induced stresses and to ensure dimensional stability. To quote from Bulletin No.S-54a on Nitronic 40 issued by ARMCO (Ref.4):

"Annealing and Stress Relieving"

"In-process annealing may be done between 1950-2050F (1066-1121C). The final annealing temperature is 1950F (1066C). Cooling practices are the same as those required for Type 304 with rapid air cooling for sheet thicknesses and water or oil quenching for heavier sections"

Nevertheless, after consultation with the supplier, LaRC chose furnace cooling after annealing because experience led to the belief that quenching would cause distortion and/or introduce quenching stresses that would in turn result in further distortion on subsequent machining. If conventional quenching media such as oil or water are used, this judgement is possibly, on balance, correct. However, despite its drastic sounding name, cryoquenching into liquid nitrogen is in fact a far less severe treatment than oil or water quenching. This is because a gas blanket is formed around the metal by the rapid and continual evaporation of liquid nitrogen caused by the large temperature difference between solid and liquid, and this gas blanket prevents liquid from coming into contact with the surface of the metal. Heat transfer across the gas blanket takes place at a steady and controlled rate, thus minimizing the possibility of quench indoced stresses. A demonstration of this effect was organized by the author during his visit to LaRC in July 1981. A Nitronic 40 aerofoil of a size suitable for the 0.3 metre cryogenic tunnel (6x8x.625in), and which had already been used by LaRC to carry out cryostability tests was used for this demonstration as its dimensions were already known to a high degree of accuracy. Remeasurement after cryoquenching showed that there were no significant dimensional changes and metallographic examination before and after heat-treatment also showed the as-quenched Nitronic 40 to be completely free from deleterious effects and to contain no carbides or sigma phase.

A thermocouple attached to the sample showed cooling from the soaking temperature of 1950F to take place initially at approximately 400F (220C) per minute and thus the sensitizing temperature range oof 1700-1100F(920-590C) was covered within a few minutes. This time is too short for significant carbide and sigma formation, but long compared with the time expected from oil or water quenches, an observation made by the technicians carrying out the quenching process.

The specimen was allowed to cool all the way down to -320F before re-warming to room temperature as this gave the most extended temperature range over which to check for dimensional changes. It would alternatively be possible to interrupt the cryoquench once the temperature of the centre of the sample had dropped below 1100F, the minimum temperature for carbide or sigma precipitation, and then allow further cooling to take place in nitrogen gas so giving a slower cooling rate over the temperature range down to ambient. Furthermore, an even more gentle heat-treatment could be envisaged in which the sample were removed from the liquid nitrogen bath once the temperature at its centre fell below 1100F, then placed in an oven at say 1000F(540C) and held at this temperature long enough to remove any residual stresses before subsequent furnace cooling to room temperature.

As noted earlier, a plate of Nitronic 40, 40in.x12in.x1.25in. thick has been successfully cryoquenched by McDonnell Douglas. Furthermore, it was intended that a specimen representative of a semi-span of a Pathfinder I wing section bolted down onto its support cradle should be subjected to a 1950F(1060C) stress relief and cryoquench cycle. This test would be as nearly representative of the full-scale wing section as could be achieved with the available Nitronic 40 and would permit an accurate assessment of the degree of dimensional stability achievable on a full scale model. Unfortunately it has not been possible to carry out this test.

Thus, if we take stock of the present position, we have the following:

1. Heat-treatment at 1950F(1060C) followed by cryoquenching has been shown to be a practical technique capable of desensitizing Nitronic 40 by removal of grain-boundary carbides and sigma phase.

2. Such treatment does not induce substantial dimensional changes or stresses: the technique is, therefore, suitable for-use at all stages prior to finish machining.

3. Further evaluation exercises are required to determine the level of residual sresses or dimensional changes before the technique can be used for a finished component. In particular, it will be essential to find out whether it is suitable for use after the 5 minute 1850F(1010C) braze cycle to be used during fabrication of the leading and trailing edges of the Pathfinder I wing.

3.5.2 <u>Intermediate temperature (1000F,530C) heat-treatments to remove machining stresses</u>

Having thus established both the undesirability of cooling slowly through the temperature range 1700-1100F(920-590C), and the technical feasibility of cryoquenching from the stress relieving temperature of 1950F(1060C) using liquid nitrogen, it is now necessary to consider the removal of any stresses induced by final machining or cryoquenching. All stress relieving treatments are rate controlled by diffusion and other mechanisms which follow the basic Arrhenius relationship,

$$\text{Rate} \propto 1/\text{time} = \text{const.} \exp.(-Q/RT),$$

where Q is the activation energy for the relevant mechanism,
R is the universal gas constant and
T is the absolute temperature.

Thus, as long as the temperature is high enough for the stress relieving mechanism to be activated, a high degree of stress relief can be attained by soaking for long times at relatively low temperatures. If the 21-6-9 Nitronic 40 type of stainless steel follows the pattern familiar for the more common 18-8 grades, adequate stress relief should be possible by soaking at temperatures of the order of 1000F(530C) for periods of the order of 10 or more hours. Slow furnace cooling from this temperature should ensure that no additional stresses are generated during cooling.

This type of intermediate temperature stress relief cycle was advocated by the writer during his visit to LaRC in July 1981 for the trailing edge of the Pathfinder wing which was due for a stress relieving heat-treatment at that time. However, it was decided to use the original Langley stress relief cycle in order to adhere to the existing time schedule for the model, but to carry out further tests to establish a schedule of times and temperatures necessary to achieve an adequate degree of stress relief from intermediate temperature heat treatments.

If intermediate temperature stress relief can indeed be shown to be effective in relieving machining stresses, then it will enable a complete schedule of desensitizing and stress relieving heat-treatments to be specified which avoid completely the problems encountered with the Pathfinder I model due to carbide and/or sigma phase formation.

4. DELTA FERRITE - ITS ORIGIN AND OCCURRENCE

4.1 Basic Principles

Delta ferrite is a body-centred-cubic phase which is stable at temperatures above about 2550F(1400C) in iron-chromium-nickel alloys, but which can also be retained to lower temperatures by high concentrations of ferrite stabilizing elements such as chromium. The presence of a few percent of delta ferrite in welds in 300 series stainless steels is often considered desirable as it helps to offset the hot cracking (slivering) that otherwise degrades welds formed in thick sections, but for cryogenic applications it is a severe disadvantage as it readily forms cleavage cracks when stressed.

One convenient way of rationalizing the stability of the phases present in stainless steels is to use the Schaeffler diagram shown in Figure 15. The chromium and nickel equivalents are calculated from the formulae:

$$\text{Cr.eq.} = (\text{Cr}) + 2(\text{Si}) + 1.5(\text{Mo}) + 5(\text{V}) + 5.5(\text{Al}) + 1.75(\text{Nb}) + 1.5(\text{Ti}) + 0.75(\text{N})$$

$$\text{Ni.eq.} = (\text{Ni}) + (\text{Co}) + 0.5(\text{Mn}) + 0.3(\text{Cu}) + 25(\text{N}) + 30(\text{C})$$

Not all the elements included in these formulae are determined in routine analyses, but substitution of the results given in Table 1 for the Nitronic 40 samples studied in this report yields the values for chromium and nickel equivalents shown in Table 9. These values are plotted in Figure 15 with the symbols as identified in Table 9, and it can be seen that all the points lie within the austenitic field, but rather uncomfortably close to the boundary with the austenite plus delta-ferrite field. In practice, it is highly likely that the various alloying elements become segregated by the casting and rolling operations and this uneven distribution can cause localized concentrations of chromium and other ferrite stabilizing elements that make the delta ferrite stable in these regions. However, the exact position of this boundary is subject to doubt and there is considerable evidence building up from recent work on high nitrogen content stainless steels to suggest that it should be moved to the left, possibly for example, to the position shown by the dotted line in Figure 15. If this were so, and the solid line were considered to be the location of the 10% delta ferrite line, it would explain the presence of the few percent ferrite found in nearly all the samples of Nitronic 40 so far examined. Furthermore, if this were so it would indicate that the delta ferrite is stable and therefore not capable of removal by heat-treatment. The results of experiments carried out to investigate this possibility are described in Section 6.

4.2 Information received on the likely processing history of Nitronic 40 supplied for use in model fabrication

Telephone conversations between the Quality Assurance Manager at G.O. Carlson's and the writer, held during his August visit to LaRC, established the likely processing history of much of the Nitronic 40 considered in this report. This history is represented schematically in Figure 16 for Heat Number 69310, cast by ARMCO and rolled to size by G.O. Carlson. The original cast billet is assumed to have solidified by dendritic growth of austenite grains from the edges which caused a segregation of alloying elements in such a way that the ferrite stabilizers were more highly concentrated at the centre of the billet. The delta ferrite thus formed would have no particular orientation but some degree of texture would be introduced during the first rolling operation in which the billet was reduced from 21 to 12 inches in thickness. At this stage it was allowed to cool to room temperature so that surface defects could be ground out (conditioning).

The slab was then reheated to 2275F(1246C) and re-rolled down to 6in. thickness x 50in. width and 100in. length. The zones of plastic work formed beneath the surface of the plate by the action of the rolls do not penetrate very far and it is unlikely that they overlapped in rolling the plate down to 6in. thickness. Thus, the delta ferrite at the centre of the plate is less orientated parallel to the rolling direction than that nearer the surfaces, an observation confirmed by metallographic examination and indicated schematically in Figure 12 for Sample D (Note that the size of the delta ferrite is greatly exaggerated to demonstrate the effect of differing orientation).

The ends and sides were cut off at this stage and the rolled surfaces ground to give the slab 5.5in. thick x 44in. wide x 60in. long subsequently delivered to LaRC and designated 69310-1F. It is not completely certain whether or not the plate was "spread", i.e. lightly cross-rolled at some stage, but the aspect ratio of the delta ferrite shown in Figure 17b) suggests that some spreading may have taken place.

Of even greater significance is the possibility that the delta ferrite is not uniformly distributed over the area of the 60in.x44in. plate. If indeed the delta ferrite was more concentrated at the centre of the cast billet, rolling would tend to push the ferrite-free edges of the casting towards the sides of the rolled slab, thereby leaving higher ferrite concentrations in the middle of the plate. Some support for this view comes from the observation that there appeared to be more delta ferrite in sample W than in either Samples S or D (see Figure 11). If this supposition is correct then the toughness indications obtained

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from the Charpy impact tests and fracture toughness measurements could give more or less conservative values compared with those governing the actual wing of Pathfinder I, depending on where the delta ferrite concentrations were greatest.

Some of the material remaining from heat 69310 after production of the 5.5in. plate was reheated and cross-rolled down from 12inches to 2inches to give the 40in.x130in. plate designated 69310-1E and subsequently delivered to LaRC. Other remnants were further reduced to a thickness of 1.25inches and designated 69310-2C. McDonnell Douglas are known to have taken delivery of one such plate, size 13in.x52in., while Lockheed Georgia received 8 plates size 14in.x60in.

Metallographic examination of three mutually perpendicular faces of a sample of the McDonnell Douglas plate, as shown in Figure 17a), revealed that the delta ferrite grains had a similar aspect ratio in both longitudinal and short transverse directions. An indication of the much higher degree of cross-rolling in these thinner plates is obtained by comparison of Figures 17a) and b). This shows the ferrite grains to be much longer and thinner (aspect ratios in excess of 30:1) in the 1.25in. thick plate, than in the 5.5in. plate (aspect ratios between 5 and 10:1).

An indication of the distribution of delta ferrite through the thickness of the 1.25in. thick plate is given in the photomontage of Figure 18. The rolled surface of the plate is located just above the first micrograph situated in the top left hand corner. A scale to the right of the micrographs gives the depth beneath the surface and about 0.25in. (7mm) is covered in each of the three vertical strips. It can be seen that the delta ferrite is more concentrated in bands located at depths of approximately 3, 4, 7, 9, 11, 12, 14 and 16mm, with a significantly smaller amount near the surface.

A slightly different ferrite morphology is displayed by the 7.5in diameter bar stock obtained for the fuselage of Pathfinder I. It is not known whether the rod was forged or drawn to shape, but whichever method was used it elongated the delta ferrite along the axis of the bar as illustrated by Figure 19. Futhermore, the amount of delta ferrite present in our sample, which is believed to have been used previously for spectographic analysis, is higher than that found in any of the samples of plate investigated. Once again, this level of delta ferrite must degrade the mechanical properties of the fuselage of Pathfinder I, particularly if it were to be subjected to shock loading.

Finally, three other samples of Nitronic 40 have been examined in this investigation. An offcut of 1in. thick plate believed to be from either heat 34794-1CI or heat 66773-2E had been obtained by the author on one of his previous visits to LaRC. This sample was coded P, and it too contained some delta ferrite as may be seen from Figure 31.

5. DELTA FERRITE - ITS EFFECT ON MECHANICAL PROPERTIES.

5.1 Scanning Electron Microscope Analysis of Charpy Fracture Surfaces containing Delta Ferrite

As noted earlier, delta ferrite has a body centred cubic structure and is therefore liable to fail by cleavage at low temperatures, particularly when subjected to shock loading. Direct evidence of such cleavage is shown in the Stereroscan views of Figure 20 which were taken from the polished and etched surface adjacent to the fracture plane in a Charpy Impact specimen tested at -320F(-196C). The low powered views in a) and b) show the narrow, oriented ferrite grains observed previously in the conventional optical micrographs, and in c)and d) the cleavage crack is shown more closely. A different ferrite grain is illustrated at x500 in Figure 20e) which has two, parallel cleavage cracks, the larger of which is shown at x1K in f) and the smaller at x2K in g). Within a given grain the cracks would be expected to be parallel because the active cleavage planes would have the same orientation. A further small crack is shown at x2K in Figure 20h).

The amount, orientation and distribution of delta ferrite within the Nitronic 40 will strongly influence its mechanical properties. Referring back to Figure 17, it can be seen that in rolled plate the ferrite is roughly disc shaped, with the plane of the disc lying parallel to the plane of the plate, and the orientation of this disc with respect to the tensile stress will have a strong influence on the mode of crack propagation. Two extreme orientations are of particular importance:

a) Disc plane parallel to tensile stress and perpendicular to crack propagation path or notch. This gives the toughest possible situation as the crack will tend to be diverted along the delaminating interface between the delta ferrite and the austenite matrix, and thus be blunted.

b) Disc plane perpendicular to tensile stress and parallel to crack propagation path or notch. This is the weakest situation as the crack will seek out the cleaved delta ferrite grains in the deformation zone at its tip and effectively jump from one already cleaved grain to the next, thus minimizing the work it has to do in shearing the tougher austenite.

The difference between these two situations can be illustrated by comparing the Charpy Impact energies of the two sets of specimens SHL and SHT (see Figure12). The SHL series, in which the delta ferrite was orientated along the bar and thus perpendicular to the notch, gave an average impact energy of 84ft.lb. at -320F, while the SHT series, in which the ferrite was perpendicular to the bar axis, gave an average of 30ft.lb!

Further clarification of this point is obtained by comparing the Stereoscan views of the fracture surface of SHT4, given in Figure 21, with those of specimen SHL1 shown in Figure 24. The orientation of the delta ferrite in SHT4 is illustrated schematically at the top left hand corner of Figure 21, together with an indication of the cleaved delta ferrite grains on the fracture surface which lie in parallel bands at 90^{o} to the notch root. The three views shown in a), b) and c) were taken at the high tilt angle of 75^{o} in order to give an appreciation of the very jagged nature of the fracture surfaces found on desensitized Nitronic 40. The remaining views d) to g) were taken with the tilt corrected and they show the parallel bands of cleaved delta ferrite in more detail. The higher magnifications show the contrast between the flat surface of the cleaved ferrite and the more distorted ductile fracture of the intervening austenite.

The differing appearance of the two modes of fracture is illustrated more clearly in Figure 23 for the partially sensitized specimen SCVL 4. Figure 23a) at x160 shows two parallel areas of cleaved delta ferrite, and in b), c)and d) the larger area on the left is shown at magnifications of x400, x800 and x1.6K respectively. The flat faceted nature of the cleavage surface is clearly visible, as is a crack lying within the ferrite along its major axis. Views e) and f) show the second smaller region of ferrite to the right of the first and here, too, the planar nature of the cleaved surface is evident. In contrast, the multiple nucleation sites of ductile fracture of the sensitized austenite are clearly revealed by the x800 and x1.6K views of g) and h) respectively.

The fracture surface of specimen SCVL9 shown in Fig. 22 differs slightly from those illustrated in Figs. 21 and 23 because the delta ferrite was more randomly oriented, although still basically lying perpendicular to the bar. The series of views a) to d) show the general appearance of the fracture surface at magnifications from x30 to x300, while e) to h) concentrate on the region at the top left corner of the area shown in a). The principal feature shown by the higher magnification views of g) and h) at x600 and x1.2K is of the cleaved delta ferrite lying at an angle to the basic fracture plane with cleavage having taken place on many different planes to give a more faceted surface than that shown for example in Fig. 23 b) to f).

The appearance of the fracture surface of sample SHL1 is markedly different as may be seen from Fig. 24. The schematic diagrams at top left illustrate the way in which the advancing crack is continually diverted along the delaminated delta ferrite interface to produce a fracture surface characterised by numerous smooth sided holes and drawn out peaks. The low magnification views of a) and b) demonstrate the large degree of deformation produced in this type of fracture, while views c),d) and e) of magnifications of x80 x160 and x400 respectively show the appearance of one of the holes in greater detail. A different part of the fracture surface is shown at x80 in f) and in g) the ductile fracture is revealed in more detail at x400.

5.2 Implications to the Pathfinder I programme of the oriented nature of delta ferrite in Nitronic 40

We have now seen that the location and orientation of the delta ferrite plays a strong influence on the resultant toughness of the material. It is fortunate, but not of course accidental, that the span of Pathfinder I wing lies along the major rolling direction of the 5.5in plate. Bending stresses generated during aerodynamic loading of the model will impose tensile or compressive stresses at the surface of the wing and any cracks trying to propagate down through the wing would be resisted by the crack-blunting action of the delta ferrite plates oriented in the plane of the wing and thus perpendicular to the crack. The toughness of the plate with respect to crack propagation in this mode is indicated by the average impact energy of 45 ft.lbs found for specimens SAOB and OC, as the were cut from material that had been through the Langley stress relieving heat treatment and were thus partially sensitized.

Cracks propagating parallel to the span of wing, or between the leading or trailing edges will however find the delta ferrite conveniently oriented to assist their propagation and for this model the toughness is indicated by the lower values of 16-17ft.lbs found for the SCV series of specimens. The likelihood or otherwise of crack propagation will of course depend on the stress levels generated during operation, and in this context it should be remembered that the model will be subjected not only to cyclic mechanical loading but also to thermal stressing during warming and cooling. At the initial materials selection stage of the wing design it had been assumed that Nitronic 40 would be capable of giving satisfactory service in this respect. Now that the extent of the degradation of the mechanical properties of Nitronic 40 due to sensitization and the presence of delta ferrite is more fully appreciated, these assumptions will have to be seriously reconsidered.

5.3 Further tests on the fracture toughness of the 5.5in thick Nitronic 40 plate

Throughout this report we have used the Charpy impact energy as an indication of the toughness of Nitronic 40; it is indeed a convenient technique for quality control and for signalling deviations from the values expected from a given grade of material. It is, however, only fracture toughness measurements that give meaningful data that can be used quantitatively to establish the relationships between critical flaw size and fracture stress. The original design criterion for the Pathfinder I model was in fact specified in terms of a minimum K_{IC} value of 85 $Ksi.in^{1/2}$ ($93.5MPa.m^{1/2}$) at the lowest operating temperature. A Charpy impact energy of 25ft.lb (34J) was taken as equivalent to a K_{IC} value of $85Ksi.in^{1/2}$ but the validity of the correlation used,

$$K_{IC} = [2E\ (C_{VN})^{3/2}]^{1/2}$$

is open to some doubt as it was established for ferrous steels in which the failure mechanism changes from shear to cleavage over the transition temperature range, and not for austenitic steels.

On realization of the extent to which the Charpy impact energies had been lowered by sensitization and the presence of delta ferrite, LaRC decided that a series of fracture toughness tests should be carried out on the 5.5in Nitronic 40 plate. Those were to include full size K_{IC} as well as compact tension specimens and be taken from material representative of the various degrees of sensitization in which this plate was known to exist. The availability of these results, together with the Charpy impact data already obtained, will in turn enable the validity of the correlation expression to be tested for sensitized austenic stainless steels.

It should, however, be noted that the remarks made earlier about the distribution and orientation of the delta ferrite also apply to the fracture toughness tests. In particular the results will have to be interpreted bearing in mind the amount and orientation of the ferrite in the zone through which the crack propagates. In this context it is believed that the fracture toughness specimens were obtained from the offcut labelled #1 in Fig. 11 and, in view of the comments illustrated in Fig.16 with respect to the distribution of delta ferrite across the area of the plate, it will be necessary to check that the ferrite distribution in offcut #1 is indeed typical of that in the critically stressed region of the Pathfinder I wing.

6. DELTA FERRITE - ITS POSSIBLE REMOVAL BY HEAT-TREATMENT

6.1 Initial heat treatments at Southampton

As noted in Section 4, all the samples studied in this programme appear to lie just inside the austenite phase field when their chromium and nickel equivalents are plotted on the Schaeffler diagram of Figure 15. If this is in fact correct, it should be possible to remove the delta ferrite by an appropriate heat-treatment that would allow diffusion and homogenization of the ferrite stabilizing elements. Accordingly, a series of heat-treatments were carried out at Southampton in August 1981 in an attempt to discover whether or not removal of delta ferrite by this technique was a practical proposition. Sample SH8 was heat-treated for 4 hours at 1830F(1000C), SH9 for 4.5 hours at 2190F(1200C) and SH10 for 0.5hr at 2000F(1100C) followed by 0.5hr at 2190F(1200C) and then 1 hour at 2370F(1300C). Metallographic sections were taken from these samples and their microstructures are shown in Figure 25 at x100 and x300, together with that of the LaRC-sensitized specimen SA. Unfortunately, there was not much delta ferrite in this part of the plate, but nevertheless the 1830F(1000C) and 2200F(1200C) heat-treatments were clearly unable to remove all of the ferrite present. Its morphology seems to have been changed somewhat by the 2200F(1200C) heat treatment in that the outlines of the ferrite grains seem to have become more rounded.

In contrast, sample SH10, which had received 1 hour at

2370F(1300C) as well as short periods at lower temperatures, definitely appeared to contain more delta ferrite after heat-treatment than before. Furthermore, its morphology had changed to a larger and more complex form. It was therefore concluded that at the higher temperature of 2370F delta ferrite actually reformed from the austenite and thus heat-treatment temperatures would have to be limited to about 2200F(1200C) if ferrite removal was to be achieved.

6.2 ARMCO work

While at LaRC in August and September 1981, the writer discussed the occurrence of delta ferrite in Nitronic 40 with the ARMCO Stainless Steel Research Laboratories. It transpired that ARMCO has a limited amount of internal research laboratory data on heat-treatment carried out on one particular batch of Nitronic 40 that contained 9% delta ferrite. A internal ARMCO report prepared in October 1976 (Ref.5) was subsequently received and it revealed that their study in fact covered the following grades of stainless steel: 301, 304, 304L, 316, 316L, 309, 302B, Nitronic 33, 40 and 50.

The work was intended to show the effect of various combinations of presoaking temperatures in the range 1800-2300F(980-1260C) and soaking at 2300 and 2380F (1260 and 1300C) on the as-cast delta ferrite structure. Changes were monitored both metallographically and by Magne-Gage (Ref.9) readings and for the particular sample of Nitronic 40 studied, complete removal of the ferrite was achieved after 4.5 hours at 2200F(1200C) or 12.5 hours at 2100F(1150C). Interestingly, at higher temperatures the ferrite started to reform. It was pointed out, however, that these results were obtained for just one laboratory melted grade, and that variations in chemical composition between different production batches would probably alter the times and temperatures required to remove the delta ferrite.

6.3 Further heat-treatments at Southampton

As noted above, the ARMCO tests were carried out on as-cast material whereas our samples have already undergone more or less extensive degrees of deformation during rolling. Thus, taking together the findings reported by ARMCO and the results of the initial heat-treatments at Southampton, it was decided to heat-treat at 2200F(1200C) as many different samples of Nitronic 40 as could be readily obtained. These samples have already been identified in Table 5 and Figures 26, 28, 29, 30 and 31 illustrate the changes in microstructure brought about by heat-treatment times of 2 and 8 hours as compared to the original structure.

Before proceeding to comment on the individual results, the strikingly obvious factor common to all these specimens is the extremely large increase in grain size brought about by the high temperature heat-treatment. These changes were measured by the technique of comparing photographs of the microstructures at x100 with a series of graded standard grain size charts indexed from

Nos. 1 to 8. For even larger grain sizes, it is necessary to make the comparisons at x50, in which case grain size numbers of 00 and 0 are reported when matched to standard chart numbers 1 and 2.

The results of these measurements are given in Table 10, and in many cases two grain size numbers are entered due to the range of grain sizes present. In the two samples taken from the 5.5in plate, D and W, the grain sizes after 8 hours at 2200F are too large even to be described by the 00 classification. This is possibly because there is less delta ferrite to restrict growth of the austenite grains, as the smallest amount of grain growth seems to have taken place in the bar sample which also had the largest concentration of ferrite.

Some appreciation of the size of these very large grains may be gained from the appearance in Figure 27 of the fracture surface of a Charpy bar cut from sample DH2 and tested at [illegible]. The impact energies obtained from these tests are also [illegible] interesting as the results given in Table 8 show. [illegible]te being quenched into liquid nitrogen after heat treatment, [illegible] therefore desensitized, the energy absorbed by the longitudinal specimens DHL1 and 2 is significantly lower than that of the corresponding desensitized, but smaller-grained, samples SHL 1, 2 and 3. In contrast, the transverse samples DHT 1 and 2 cut from the large grain material are tougher than the corresponding small grain samples SHT 1, 2 and 3. Furthermore, despite the liquid nitrogen quench after heat-treatment, the fracture surface shown in Figure 27 has a highly intergranular look about it, a characteristic not usually expected from a desensitized austenitic steel.

Magne Gage measurements were also made on this series of samples in order to give an independent indication of the amount of delta ferrite present. These results are given in Table 11 in terms of ferrite number, which for the levels concerned are equivalent to volume percentage of ferrite. Many of the readings are at the limit of sensitivity of this technique which is about 0.1 and when carrying out the measurements it was also noticeable that at ferrite numbers of less than 0.5 the readings varied with position, probably a reflection of the scattered distribution of the delta ferrite in these almost fully austenitic samples. It can be seen that the Magne Gage readings support the conclusions already drawn from the metallographic examination. In particular, samples S and D both were shown to contain only about 0.3% ferrite in the as-received condition, although the desensitized Charpy bars seem to have been cut from an area with about 0.4 to 0.5% ferrite. There was a small decrease to 0.22% after the 8 hour treatment at 2200F but a significant rise to 1.36% as a result of the 2370F treatment in which the metallographic results had suggested reformation of the delta ferrite.

The comment made earlier about the higher ferrite content of the W specimens taken from near the leading edge of the Pathfinder wing is also confirmed by the Magne Gage readings, which remain high even after the 2200F heat-treatment. Both the McDonnell Douglas 1.25in plate and the Langley 1.0in plate have low ferrite contents similar to those of Samples S and D, and the 2200F heat-treatments seem to cause a slight decrease in ferrite number. The metallographic observations on the bar Sample B are also confirmed by the Magne Gage readings which show the highest percentage of delta ferrite in any of our samples, 2.5 to 4.7%. Heat-treatment at 2200F seems to cause a definite decrease, but even after the 8 hour treatment, there was still nearly 3% ferrite present.

The ease with which Magne-Gage readings can be obtained suggests that it would be worth carrying out a magnetic survey of the various parts of the Pathfinder 1 model to determine how much delta ferrite is where. Given the unlikelihood of being able to remove the ferrite by heat-treatment it should at least be worthwhile finding out where it is most concentrated and hence likely to cause most harm.

6.4 Current position

The practical implications of the results discussed so far in this section are that it is going to be extremely difficult, if not completely impossible, to remove the delta ferrite from the existing stocks of Nitronic 40 currently held a LaRC and other organizations building models for cryogenic wind tunnels. Furthermore, any such heat-treatments have to take place at temperatures which cause very extensive grain growth. The justification for further work in this direction will, however, depend on whether it is considered that meterial with such large grains would be generally acceptable for use. For example,

- will it machine satisfactorily?
- will it be dimensionally stable on cryocycling into liquid nitrogen?
- will the thermal expansion cause anisotropic changes in dimensions that would interfere with the aerodynamic measurements?
- will the mechanical properties be any better than those of the existing material?

Instinctively, one feels that there must be problems with such very large grained material and, even if there are no serious problems, will the advantages be sufficient to justify the considerable effort and expense involved? While presuming that the answer to this question will be no, it is probably worthwhile carrying out some further tests on this material to find out more about its machinability, dimensional stability and mechanical properties. Accordingly, a sample about 2in. x 2in. x 5.5in. thick is being returned to LaRC for further tests.

7. RECOMMENDATIONS CONCERNING THE ACCEPTABILITY OR OTHERWISE OF MODEL COMPONENTS ALREADY BEING MANUFACTURED FROM DEGRADED NITRONIC 40

Regardless of what may be theoretically possible from an appropriate series of heat-treatments, it is accepted that the final decisions have to be made in an engineering environment and that it is therefore a realistic, balanced judgement that is being sought for the Pathfinder I programme. Nevertheless, in any cryogenic load bearing application, it is axiomatic that toughness must be maximized as far as possible in order to protect both life and the structure concerned. In the case of the Pathfinder 1 model, its operation at the lowest design temperature with the Nitronic 40 in such a severely degraded state would pose a serious risk of damage to the model, and more importantly to the NTF tunnel itself. At this late stage, it appears that there are relatively few options open and the following are some of the arguments for and against the more obvious possibilities.

7.1 Run the model at temperatures above the minimum design point

As the toughness of most sensitized austenitic stainless steels with and without delta ferrite decreases at lower temperatures, operation should be adequately safe at some temperature below ambient that would have to be determined in the light of more detailed knowledge of the temperature-toughness relationship. This would allow experience of general operating procedures to be built up and also should permit valid aerodynamic data to be obtained for this restricted temperature range.

Against this are the disadvantages of not being able to explore the full temperature and pressure limits of the NTF design. Furthermore, in the event of a tunnel malfunction that resulted in exposure of the model to temperatures close to that of liquid nitrogen, a dangerous situation could develop if the embrittled model could not be rapidly unloaded.

7.2 Heat-treat the model to maximize its toughness

From the detailed evidence presented in the earlier sections, it is obvious that there are two basic metallurgical factors responsible for the present degraded state of the model material: sensitization and delta ferrite.

In the latter case, it is, in our view, unrealistic to expect to be able to effect any substantial changes in delta ferrite levels without incurring unacceptably large increases in grain size, and also possibly machining and dimensional stability problems. It is therefore recommended that such heat-treatments are discounted as unrealistic.

On the other hand, we believe that it is possible to enhance significantly the toughness of the material, to a level in fact higher than the minimum acceptance criterion originally chosen for NTF models. This would involve a combination of solution heat-treatments at 1950F(1065C) followed by cryoquenching in liquid nitrogen to prevent carbide and sigma phase formation, and post machining stress relief for long periods in a vacuum or inert gas shielded furnace at 1000F(530C).

It is not yet too late to adopt this philosophy and we believe that would offer the best way out of the present difficulty.

Arguments against this philosophy include the following:

- it is too late to change the schedule: in our opinion, later and better is to be preferred to "on time and degraded".

- cryoquenching might cause warpage: it might, but if done early enough in the machining cycle it can be corrected. Also, it could be argued that cryocycling between room and the lower operating temperatures could also cause thermal distortion. If so, it is better that such distortion is created deliberately at an early stage so that it can still be remedied.

- it might not be possible to obtain adequate post-machining stress relief with the 1000F(530C) temperature cycle: possibly so, but only realistic tests will prove the point. Furthermore, it would probably be possible to raise the temperature to about 1050F(566C) and still not precipitate carbides during holding times about 10-12 hours.

Considering the various components of the Pathfinder I model separately, it is the wing that will experience the highest aerodynamic loadings. It is therefore in our opinion particularly important that this material be given the maximum toughness possible. If the body is less heavily stressed it might be acceptable to leave it un-heat-treated. However, the round bar from which the body was machined had the highest delta ferrite content of all the Nitronic 40 samples investigated, and there does not seem to be any Charpy impact data that would give an indication of the toughness of the material in its stress-relieved state.

7.3 Establish experimentally the fracture toughness of the degraded material

In section 5.3 it was noted that an experimental programme had been set up at LaRC to determine the fracture toughness of the degraded Nitronic 40 being used for the Pathfinder I wing, and initial results suggest that the toughness is significantly higher than the minimum value required for models in the NTF. A thorough fracture mechanics analysis and non-destructive testing programme will also be carried out to determine the relationship between working stress, toughness and acceptable flaw size.

8. SPECIFICATION AND PURCHASE OF MATERIALS FOR CRYOGENIC WIND TUNNEL APPLICATIONS

8.1 Introduction

Most materials are normally supplied to specifications that are based on either chemical composition, mechanical properties or both. Additional restrictions are often imposed to meet more specific requirements such as hardenability, weldability and free machinability, or increased yield stress, formability or corrosion resistance. Sometimes these additional requirements are indicated by different type numbers, as in the 321 and 347 types of stainless steel which contain titanium and niobium to prevent weld decay, or the molybdenum containing type 316 which is most resistant to corrosion in sea water. Alternatively, an additional qualifying letter is added to the basic type as in the low carbon (<.035%) 304L, or the high proof (.2% nitrogen) 316N, grades of stainless steel.

The Nitronic 40 examined in this investigation met the chemical composition specifications, as indicated in Table 1, yet it contained significant quantities of delta ferrite that severely limits its use in the application for which it was purchased. It is therefore, in our opinion, necessary to formulate more closely the specifications under which materials are purchased for cryogenic wind tunnel applications. Although the details of the following recommendations are intended to apply specifically to Nitronic 40, the spirit should also be considered more generally for materials used in this most demanding application.

There are numerous precedents for laying down additional requirements for more exacting applications, particularly in aerospace and military applications. There is also a cryogenic precedent in the E.L.I. (Extra Low Interstitial) grades of Titanium alloys that had to be developed in order to overcome the brittleness which previously had made Titanium alloys unsuitable for cryogenic use.

8.2 Suggested Criteria for Nitronic 40

(a) Chemical

This can remain unchanged.

(b) Mechanical

This should include realistic values for the yield and ultimate tensile stresses and percentage elongation at both room temperature and -320F. <u>It is also vital that a Charpy Impact energy is specified for a test at -320F.</u> This value should be set high enough to detect degrading characteristics such as sensitization and delta ferrite and should be of the order of 65ft.lb, although more work needs to be carried out before an exact value is specified.

(c) Metallurgical Structure

There should be a very low figure put on the maximum amount of delta ferrite acceptable regardless of whether the chemical and mechanical specifications are met. Metallographic techniques of detection are sensitive down to levels of <1% but at these limits the problem becomes somewhat statistical in nature and depends on how representative the samples are of the rest of the material. Metallographic techniques are also capable of detecting carbide and sigma phase precipitation and would offer a cross-check on the impact test results.

(d) Magnetic

Magne Gage and other magnetic measuring techniques are capable of detecting delta ferrite and other ferro magnetic phases present down to levels of about 0.1 to 0.2%. They are also quick and cheap and therefore capable of use for routine quality control work to check material at the "as-received" stage.

8.3 Implementation

Although we have no figures on hand to prove the point, it is highly probable that in most applications related to cryogenic wind tunnels, the cost of the basic metal is a small fraction of the total cost. If this is indeed the case, then additional charges necessary to ensure that a project starts off with premium quality material would be a worthwhile insurance policy to prevent subsequent losses and delays, or even the possible need to restart the whole project.

The following measures are suggested in order to help implement a policy of purchasing top quality materials for the cryogenic wind tunnel model programme:

1) If possible the purchase of such materials should be co-ordinated by a small group that has adequate knowledge and technical back-up for the task.

2) The person(s) designated should build up contacts at a high technical and sales level in suppliers such as ARMCO, CARPENTER, US STEEL, etc. When material is required, these contacts should be told in detail the application for which the material is required and any special problems that might arise. These requirements, and the offers and technical advice received, should be written down and filed. Agreement in writing should be obtained for supplying to the criteria indicated above.

3) Documentation relating to purchase of material should specify the use to which it will be put and the criteria by which it will be accepted.

4) The material should be finally accepted only after the criteria listed above in Section 8.2 have been checked either by the purchaser or by an approved independent test house.

5) It should be clearly recognised that such a policy will probably make some potential suppliers unwilling to tender for material and that those who do will expect a premium price for material of such proven quality. It is, however, our belief that this additional money would be very well spent and go a long way towards avoiding the problems that have already arisen with the existing Nitronic 40.

9. THE USE OF NITRONIC 40 IN OTHER CRYOGENIC APPLICATIONS

The driving forces behind the advances in understanding of the properties of materials at cryogenic temperatures that have taken place over the last few decades, have come from a numb.r of differing technological projects including nuclear weapons, cryogenically fuelied rockets, high energy nuclear physics and liquefied natural gas transport and storage. At present the impetus for further developments appears to come largely from the field of nuclear fusion and the need to build very large superconducting magnets to contain and shape the nuclear plasma.

Many of the properties required of materials utilised in these magnets are similar to those needed for cryogenic wind tunnel applications, in particular the need for high strength and toughness at the low operating temperatures, although they do not, in general, require the very fine surface finishes demanded of aerodynamic models. Most of the materials evaluated for the construction of Pathfinder I were also considered for these projects and in at least two cases, the Westinghouse Large Coil (Ref. 7) and the Lawrence-Livermore MFTF Magnet (Ref. 8), Nitronic 40 was the chosen material. In both cases, however, problems arose during fabrication which resulted in the abandonment of Nitronic 40 and the substitution of alternative materials.

In the case of the material used for sheathing the superconducting windings, the requirements differed from those needed for wind tunnel models in that the sheath was made from 1.6mm thick Nitronic 40 autogeneously G.T.A. welded to form a pressure tight container around the conductors. The whole conductor assembly was then coiled and heat-treated at 700C(1300F) for 30 hours to form the superconducting Nb_3Sn compound by a reaction anneal. A severe loss of ductility resulting from this heat-treatment was found to be due mainly to the conversion of delta ferrite in the weld zone into sigma phase and austenite, but the parent metal was also significantly degraded. These findings match very closely the results outlined in Section 3.3 of this report.

Other applications in which the Nitronic 40 was to be used in thicker sections also ran into problems due mainly to the presence of delta ferrite. Further details of these cases are still being gathered and will be available for a future report.

One other significant trend worth noting in this context is the large amount of work taking place both in the U.S.A. and Japan on the development of other high manganese high-nitrogen stainless steels. The higher manganese levels are needed to improve the formability of these steels that would otherwise be impaired by the high nitrogen levels added to improve the proof stresses. These developments should be monitored closely to see whether they do eventually result in commercially available quantities and product forms likely to be of interest for use in the cryogenic wind tunnel programme. Nevertheless, one of the major lessons to be learned from the problems encountered with Pathfinder I is the need for very careful specification and quality control of the production material to ensure that it also achieves the mechanical properties demonstrated by the pre-production material and quoted in the manufacturers technical literature.

Finally, it is also worth noting that in a number of cases, the precipitation-hardenable alloy A286 was used as a replacement for Nitronic 40 in the programmes noted above. This alloy has given very satisfactory service when used for airfoil models in the 0.3 metre Transonic Cryogenic Tunnel and its use for future NTF models must be very seriously considered.

10. CONCLUSIONS

1. A comprehensive study has been carried out to investigate the metallurgical characteristics of Nitronic 40 material in connection with its use in Cryogenic Wind Tunnel Models. In particular, the effects of carbide and sigma phase precipitation resulting from heat treatment, and the presence of delta ferrite are evaluated in relation to their effects on mechanical properties and the potential consequences of such metallurgical degradation.

2. Methods were examined for desensitizing the material and for possible removal of delta ferrite as a means for restoration of the material to its advertised properties. It was found that heat treatment followed by cryoquenching is a technique capable of desensitizing Nitronic 40. However, it was concluded that it is extremely difficult, if not impossible, to remove the delta ferrite from the existing stock of material.

3. The implications of using the degraded Nitronic 40 material for cryo model testing are reviewed and recommendations are submitted with regard to acceptability of the material.

4. The experience gained from the Nitronic 40 study clearly identifies the need to implement a policy for purchasing top quality materials for cryogenic wind tunnel model applications. Also the study exemplifies the need for careful evaluation and analysis of processes used in the fabrication of metallic alloys for cryogenic use.

December 1981.
Amended Version March 1982

DAWigley
D.A. Wigley

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TABLE 1
Chemical Composition of Nitronic 40 Samples

Identification	C	Mn	P	S	Si	Cr	Ni	N_2	Other
Armco Data UNS S21900	.08 max	8.0/ 10.0	.06 max	.03 max	1.0 max	19.0/ 21.5	5.5/ 7.5	.15/ .40	-
UNS S21904	.04 max	"	"	"	"	"	"	"	-
69310-1F,-1E, and 2C mill certs.	.026	9.30	.021	.003	.67	20.32	6.68	.29	-
LaRC anal. of 69310.	.032	11.84	-	.003	-	20.45	6.73	-	-
306535	.023	9.08	.028	.002	.67	20.41	7.14	.25	.24Mo .30Cu
66773-2E	.030	8.97	.013	.009	.53	19.74	6.50	.26	-
34794-1C1	.017	8.83	.030	.004	.67	20.37	7.08	.34	-

TABLE 2
Typical Mechanical Properties at Cryogenic Temperatures* of
Nitronic 40, 4.75"(118mm) Thick Slab - Annealed.
(Armco Bulletin No.S-54a)(Ref.3)

Test Temp. F(C)	UTS ksi (MPa)	0.2% YS ksi (MPa)	Elong. % in 1" (25.4mm)	R/A %	Charpy Impact V-notch ft-lbs(J)
75 (24)	103 (710)	58 (400)	50	70	205 (294)
-110 (-79)	134 (924)	87 (600)	59	71	146 (196)
-320 (-196)	203 (1400)	150 (1034)	-	24	65 (87)
-423 (-253)	245 (1689)	196 (1351)	15	20.5	53 (71)

*Tested in transverse direction.

TABLE 3
Schedule of Temperatures and Times for Carbide Formation in Nitronic 40 (Armco Bulletin No. S-54a)

Temperature F(C)	Carbide Formation Rating* Time, Hours 1	9	25	100
1000 (538)	None [1]	None [1]	None [1]	None [1]
1100 (593)	None [1]	Trace [1.6]	Light [3.2]	Heavy [5]
1200 (649)	Trace [1.6]	Medium[3.6]	Heavy [4.8]	V.Heavy[5.6]
1400 (760)	Light [3]	Light [3]	Medium[3.8]	Medium[3.8]
1600 (871)	Trace [1.6]	Trace [2]	Trace [2]	Trace [2.5]

* Carbide Rating - Solar Aircraft Co., San Diego, California.
None - (1 to 1.4)
Trace - (1.5 to 2.5)
Light - (2.6 to 3.4)
Medium - (3.5 to 4.5)
Heavy - (4.6 to 5.4)
V.Heavy - (5.5 to 6).
Numbers in [] are average Solar Carbide Ratings of 5 heats of Nitronic 40 Stainless - 0.040% max. carbon.

TABLE 4
Schedule of Temperatures and Times for Sigma Phase Formation in Nitronic 40 (Armco Bulletin No.S-54a)

Temperature F(C)	Sigma Formation Rating* Time, Hours 1	9	51	100
1000 (538)	None [1]	None [1]	None [1]	None [1.2]
1100 (593)	None [1.2]	None [1.2]	None [1.2]	Trace [1.6]
1200 (649)	None [1.2]	None [1.2]	Trace [2]	Light [2.6]
1400 (760)	None [1.4]	Light [2.8]	Light [3.4]	Medium[3.6]
1600 (871)	Trace [1.6]	Trace [2.4]	Light [3]	Light [3.4]

*Sigma Rating
None - (1 to 1.4)
Trace - (1.5 to 2.5)
Light - (2.6 to 3.4)
Medium - (3.5 to 4.5)
Heavy - (4.5 to 5)
Numbers in [] are average rating of 5 heats of Nitronic 40 - 0.040% max. carbon.

TABLE 5 : IDENTIFICATION OF NITRONIC 40 SAMPLES REFERRED TO IN THIS REPORT

Code	Heat No.	Origin/Description	Heat-treatment
SA	69310-1F	2.5in.x1.25in.x5.5in.thick sample from 5.5in. plate obtained July 1981.	LaRC HT cycle of hold at 1950F(1065C) then furnace cooled over many hours.
SCV	69310-1F	Charpy Test samples.	As above.
SH1-7	69310-1F	Specimens cut from SA	Desensitizing cycle of 30 minutes at 1950F (1065C) then liquid nitrogen quench.
SH8-10	69310-1F	Specimens cut from SA	HT at and above 2000F (1100C) to modify delta ferrite.
ST	69310-1F	Specimens cut from SA	HT at 1380F(750C) to induce carbide and sigma formation.
DA	69310-1F	Wedge shape 4.5in.x2.5-0in x5.5in.thick. Sample obtained September 1981.	As delivered from G.O. Carlson.
DH	69310-1F	Specimens cut from DA	HT at 2200F(1200C) to modify delta ferrite
WA	69310-1F	4.5in.x1.25in.x1in.offcut from leading edge of left wing of Pathfinder I, obtained September 1981.	LaRC HT cycle of hold at 1950F(1065C) then cool thinner section in 2-3 hrs?
WH	69310-1F	Specimen cut from WA	HT at 2200F(-ferrite)
MA	69310-2C	Approx. 2.5in.x1in.x1.25in. thick plate sample obtained from McD/D Aug.'81.	As delivered from G.O. Carlson
MH	69310-2C	Specimens cut from MA	HT at 2200F(-ferrite)
LA	69310-2C	Charpy specimens from similar plate supplied to Lockheed.	
BA	306535	Sample approx.3in.x3in.x 0.5in. cut from 7.5in.dia. barstock.	As delivered from G.O. Carlson.
BH	306535	Specimens cut from BA	HT at 2200F(-ferrite)
PA	34794-ICI or 66773-2E	Sample approx.2.25in.x 1.5in.x1.0in.thick plate obtained January 1981.	Presumed as-received
PH	"	Specimens cut from PA.	HT at 2000F(-ferrite)
TA	-	Small sample from 6in.x 8in.x0.625in.dimensional stability aerofoil.	30 minutes at 1950F (1065C) then liquid nitrogen quench.

TABLE 6
MICROPROBE ANALYSIS OF AUSTENITE AND DELTA FERRITE IN NITRONIC 40

Phase	Chromium	Nickel	Iron + other
Austenite	17.7	7.3	75.0
Delta Ferrite	22.9	3.4	73.7
Macro analysis	20.4	6.7	72.9

TABLE 9
CHROMIUM AND NICKEL EQUIVALENTS FOR NITRONIC 40 SAMPLES

Sample Heat No.	ARMCO tests	69310 Ladle	306535	66773	34794
Cr.Eq.	22.3	21.6	22.1	20.8	21.7
Ni.Eq.	20.0	19.4	18.7	18.9	20.5
Symbol in Fig.15.	×	●	▼	■	◉

TABLE 7 : CHARPY V-NOTCH IMPACT ENERGIES FOR NITRONIC-40 SPECIMENS TESTED BY LaRC AT CRYOGENIC TEMPERATURE

Source	Specimen Identification.	Bar Orientation w.r.t. Rolling Direction.	Condition or Treatment	Impact Energy (ft.lb)
BILLET	SCVL 1	THROUGH THICKNESS	Stress relieving	25
69310-IF	SCVL10	notch parallel	(SR) cycle of 1hr	21
5.5in.	SCVL 6		at 1950F (1065C)	19
PLATE	SCVT 1	notch at 90°	then furnace	21
P.F.1	SCVT12		cooled.	20
WING	SCVT 6			19
Note:	SCVL 4	notch parallel	One SR cycle plus	27
This	SCVL 8		simulated braze, 5min.	25
series of	SCVL10		at 1850F(1010C)	24
samples				
tested at	SCVT 8	notch at 90°	Two SR cycles plus	31
-275F	SCVT 4		one simulated braze	20
July'81.	SCVT10		cycle as above.	19
Tested	SCVL 2	notch parallel	One SR cycle of 1hr	21
Oct.'81	SCVL 5		at 1900F(1040C) then	17
at -320F	SCVL 9		furnace cooled, followed	15
			by cryocycling between	
	SCVT 5	notch at 90°	+60F(20C) and -320F(196C)	-
	SCVT 9			14
BILLET	LLS 1	PARALLEL	(presumed similar to	43
69310-2C	LLS 2	notch at 90°	LaRC SR cycle)	37
1.25in.	LLS 3			39
PLATE	LLT 1	notch through-		42
used for	LLT 2	thickness		39
Lockheed	LLT 3			38
Georgia	LLT 4			39
model.	LLT 5			33
Tested	LLT 6			38
Oct.'81	LTS 1	TRANSVERSE		40
	LTS 2	notch parallel		33
	LTS 3			37
	LTT 1	notch through-		34
	LTT 2	thickness		32
	LTT 3			34
#3		PARALLEL		
Dimens.	T3 LS	notch at 90°	H.T. at 2000F(1100C)	79
stability	T3 LT	" through	followed by air cooling,	70
6in.x6in.		TRANSVERSE	ground flat, cryocycled	
x0.625in.	T3 TS	notch parallel	to -320F, HT 30mins. at	85
Aerofoil.	T3 TT	" through	1950F(1065C) then	80
			quenched in L.N.	

TABLE 8 : CHARPY V-NOTCH IMPACT ENERGIES FOR NITRONIC-40 SPECIMENS TESTED AT SOUTHAMPTON UNIVERSITY at [illegible](-196C)

Source	Specimen Identification.	Bar Orientation w.r.t. Rolling Direction.	Condition or Treatment	Impact Energy (ft.lb)
BILLET	SHL 1	PARALLEL	LaRC SR cycle then	83
69310-IF	SHL 2	notch at 90°	desensitizing HT of 30	88
Desensi-	SHL 3	"	mins at 1950F(1050C)	82
tizing	SHT 4	THROUGH	then quenched in	32
H.T.	SHT 5	THICKNESS	liquid nitrogen.	27
Aug.1981	SHT 6	notch at 90°		31
		PARALLEL		
As	SA 0B	notch at 90°	LaRC SR cycle of 1950F	41
received	SA 0C	" through	then quenched in L.N.	48.5
BILLET	ST 5B	" at 90°	SR plus 5hr at 1380F	21
69310-IF	ST 5C	" through	then quenched in L.N.	24
Sigma	ST 24B	" at 90°	SR plus 24hr at 1380F	12.5
Sensitiz-	ST 24C	" through	then quenched in L.N.	12.0
ing	ST 72B	" at 90°	SR plus 72hr at 1380F	7.1
Treatment	ST 72C	" through	then quenched in L.N.	6.9
Nov.1981.	ST168B	" at 90°	SR plus 168hr at 1380F	4.5
	ST168C	" through	then quenched in L.N.	4.6
		TRANSVERSE		
BILLET	DHL 1	Notch parallel	Heat-treated for 8 hrs	55
69310-IF	DHL 2	Notch through	at 2200F(1200C) then	41
Delta		thickness	quenched in Liquid	
Ferrite			Nitrogen.	
Removal		THRU-THICKNESS		
H.T.	DHT 1	Notch at 90°		33
Oct.1981.	DHT 2	Notch parallel		43
ARMCO DATA		PARALLEL	Annealed.	112-118
			H.T. 2hrs at 1250F(677C)	
			to simulate HAZ in welds.	20-21

Note: 1ft.lb = 1.356 Joules.

TABLE 10
ASTM GRAIN SIZES FOR HEAT-TREATED NITRONIC 40 SAMPLES

Sample \ Condition	As-Rec'd.	2hrs @ 2200F	8hr @ 2200F	0.5hr @ 1950F	0.5hr @ 2000F + 0.5hr @ 2190F + 1hr @ 2370F.
S	3-2	-	(4.5hr) 00	3-2	1
D	3-2	1-0	<00	-	-
W	2	0	<00	-	-
M	2-1	1-0	00	-	
P	2	1	1-0	-	-
B	3-2	2-1	1	-	-

TABLE 11
MAGNE GAGE MEASUREMENTS ON AS-RECEIVED AND HEAT-TREATED SAMPLES OF NITRONIC 40. ENTRIES GIVEN AS FERRITE NUMBER (% FERRITE)

Sample \ Condition	As-Rec'd.	2hrs @ 2200F	8hrs @ 2200F	0.5hr @ 1950F	0.5hr @ 2000F+ 0.5hr @ 2190F+ 1hr @ 2370F
D	.29,.24	.18	0	-	-
S	.32	-	(4.5hr) .22	.51,.41,.46 .38,.4,.4	1.36
W	1.45,1.77	1.92	1.13	-	-
M	.18,.22	.09	.18	-	-
P	.21,.09	.05	.06	-	-
B	4.73,4.32 3.79,2.54	3.85	2.96	-	-

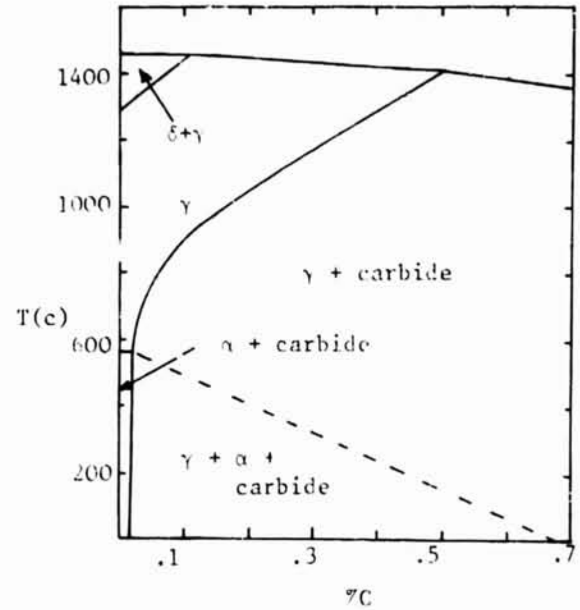


FIG.1: PHASE DIAGRAM FOR 18Cr-8Ni STAINLESS STEEL

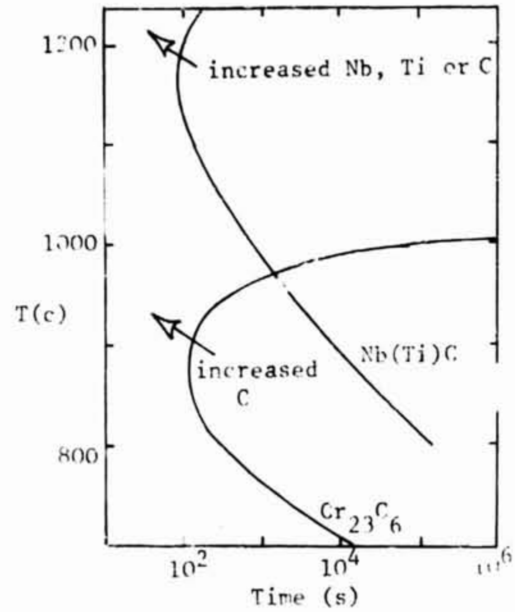


FIG.2: T.T.T. CURVES FOR GROWTH OF $M_{23}C_6$ AND Nb(Ti)C in Cr-Ni STAINLESS STEELS.

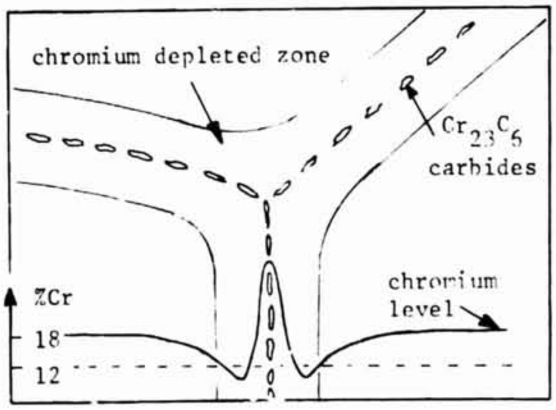


FIG.3: SCHEMATIC REPRESENTATION OF CARBIDE PRECIPITATION AND CHROMIUM DEPLETION IN 18/8 STAINLESS STEEL

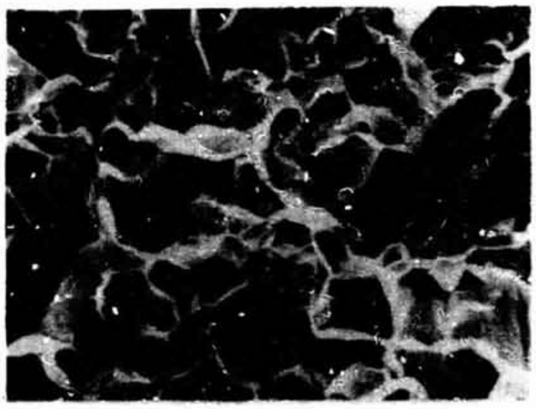
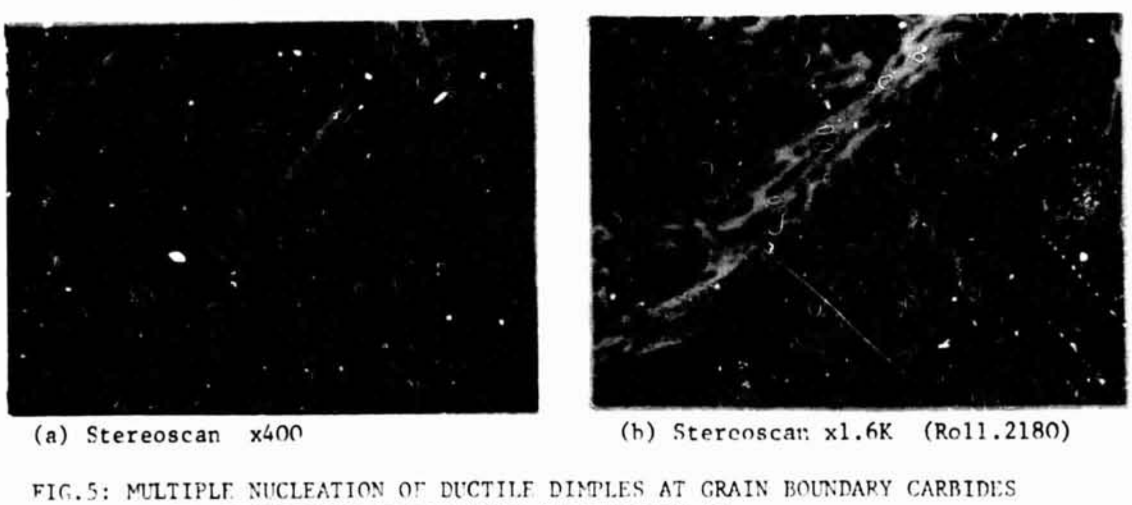


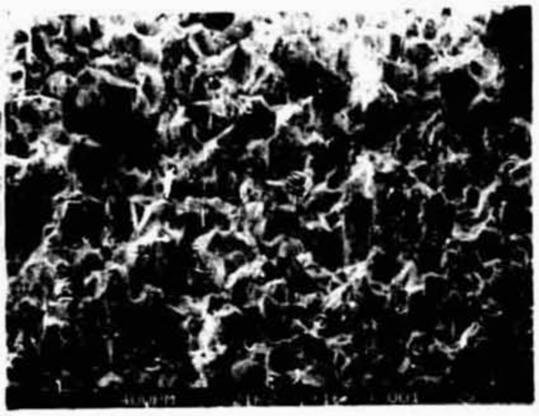
FIG.4: STEREOSCAN VIEW AT x400 OF INTERGRANULAR FRACTURE IN 18/8 STAINLESS STEEL (NN.1026).



(a) Stereoscan x400

(b) Stereoscan x1.6K (Roll.2180)

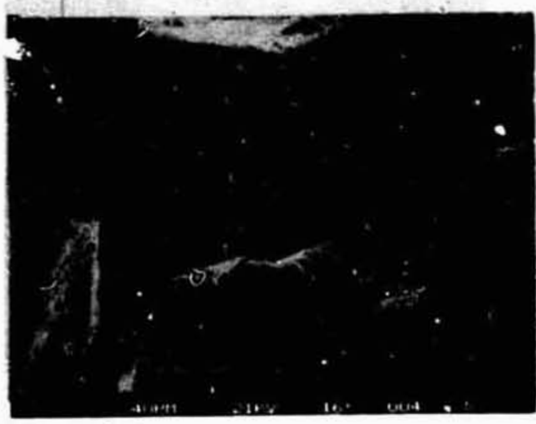
FIG.5: MULTIPLE NUCLEATION OF DUCTILE DIMPLES AT GRAIN BOUNDARY CARBIDES DURING INTERGRANULAR FRACTURE OF NITRONIC 40.



a) x 30

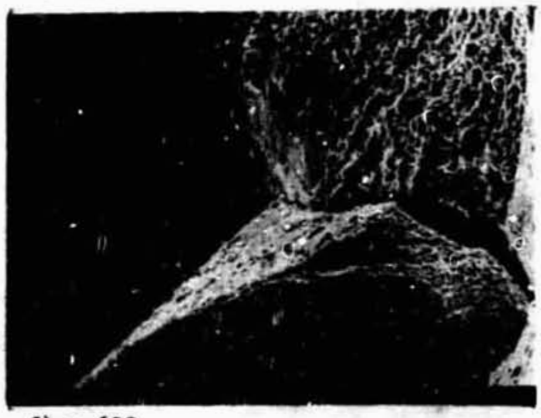
b) x 60

c) x 120

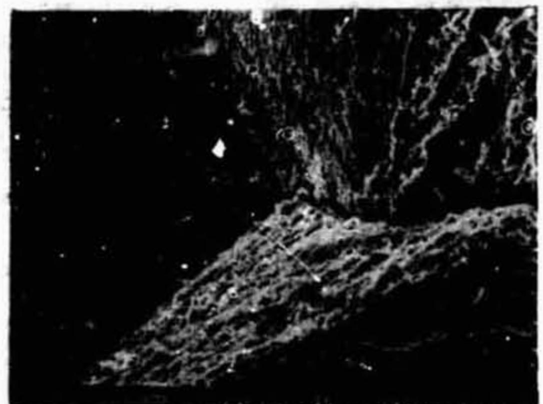


d) x 300

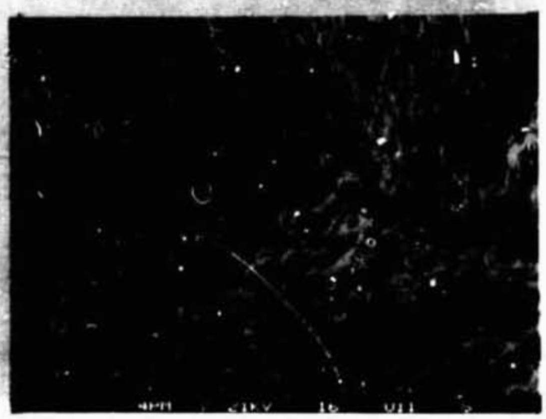
e) x 300



f) x 600



g) x 1.2K



h) x 3K

FIG.6: STEREOSCAN VIEWS OF -320F CHARPY FRACTURE SURFACE OF HIGHLY SENSITIZED NITRONIC 40. SPECIMEN ST168 (Roll No.2207).

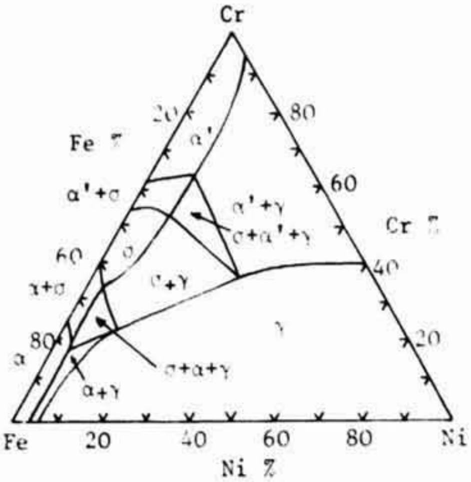


Fig. 7. Fe-Cr-Ni Ternary Diagram at 1470F (800C)

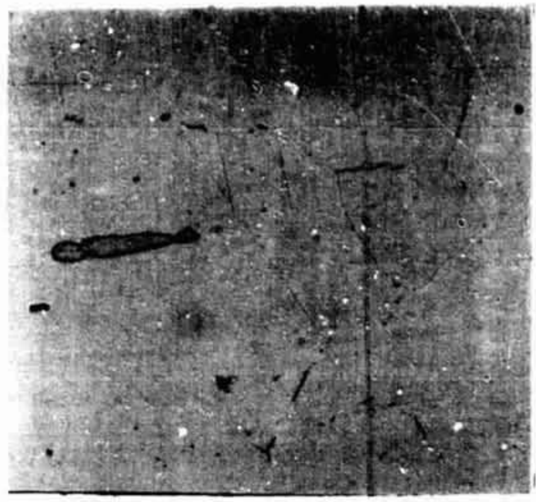


Fig. 8. a) Langley HT, SA, x300, NN.2187

b) Langley HT +5hrs, x300, NN.2174

c) Langley HT +24hrs, x300, NN.2177

d) Langley HT +72hrs, x300, NN.2184

e) Langley HT +168hrs, x300, NN.2194

FIG. 8. DEVELOPMENT OF CARBIDES AND SIGMA PHASE DURING HEAT-TREATMENT AT 1380F (750C) IN SAMPLE S.

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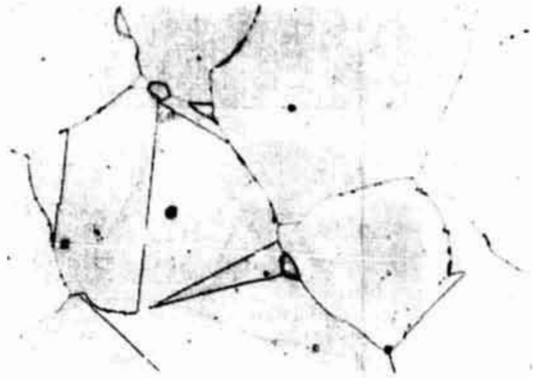
a) Desensitized SH7, x300, NN.2045.



b) As delivered, DA, x300, NN.2110.

c) Langley HT, SCVT4, x300, NN.2020.

d) Langley HT, WA, x300, NN.2168.

e) McD-D, MA, x300, NN.2166

f) Lockheed, LA, x300, NN.2204.

g) Langley HT, SA, x600, NN.2189.

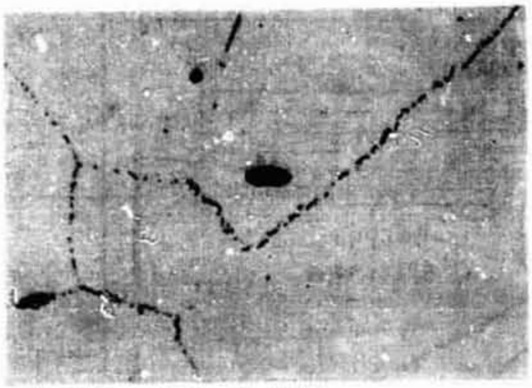
h) Langley HT, SA, x600, NN.2188.

FIG. 9: DEGREE OF SENSITIZATION PRESENT IN VARIOUS SAMPLES OF NITRONIC 40, 20 sec. MIXED ACIDS ETCH

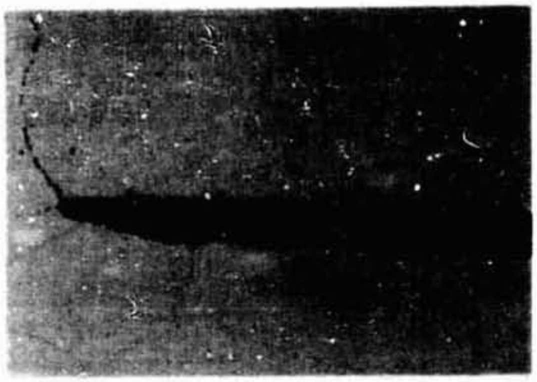
BLACK AND WHITE PHOTOGRAPH

a) ST5, x600, NN.2176.

b) ST5, x600, NN.2175



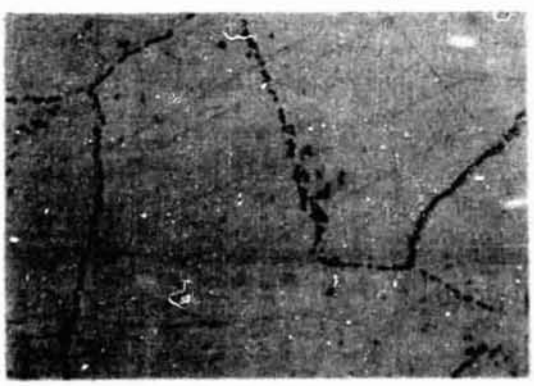
c) ST24, x600, NN.2179.



d) ST24, x600, NN.2178.

e) ST72, x600, NN.2186.

f) ST72, x600, NN.2185.



g) ST168, x600, NN.2196.

h) ST168, x600, NN.2195.

FIG.10: DEVELOPMENT OF SIGMA PHASE PRECIPITATES AT GRAIN BOUNDARIES AND WITHIN DELTA FERRITE DURING HEAT-TREATMENT AT 1380F (750C) IN SAMPLE S.

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OF POOR QUALITY

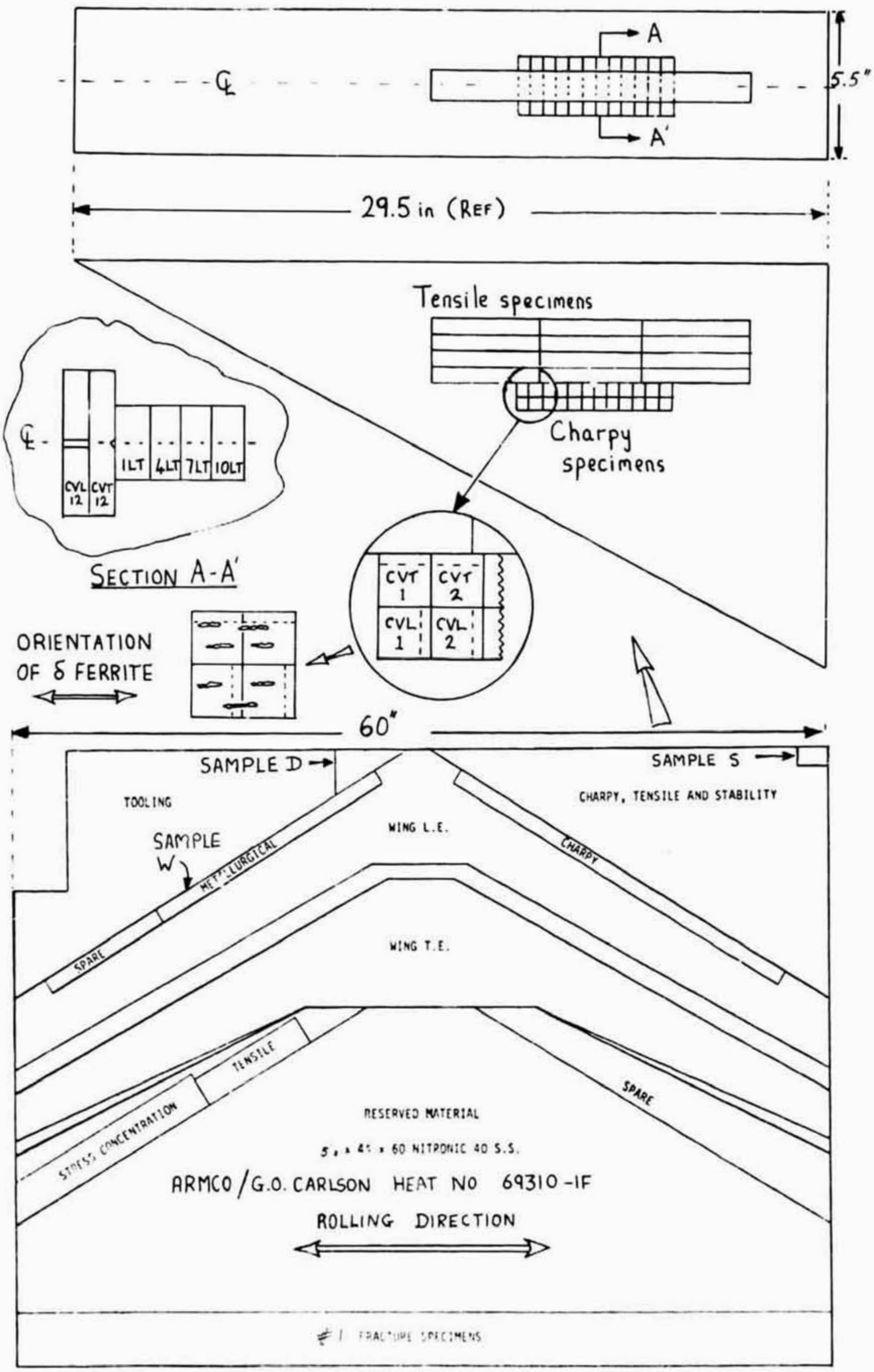


FIG.11: LOCATION OF CHARPIES, TENSILES AND SAMPLES D, S and W.

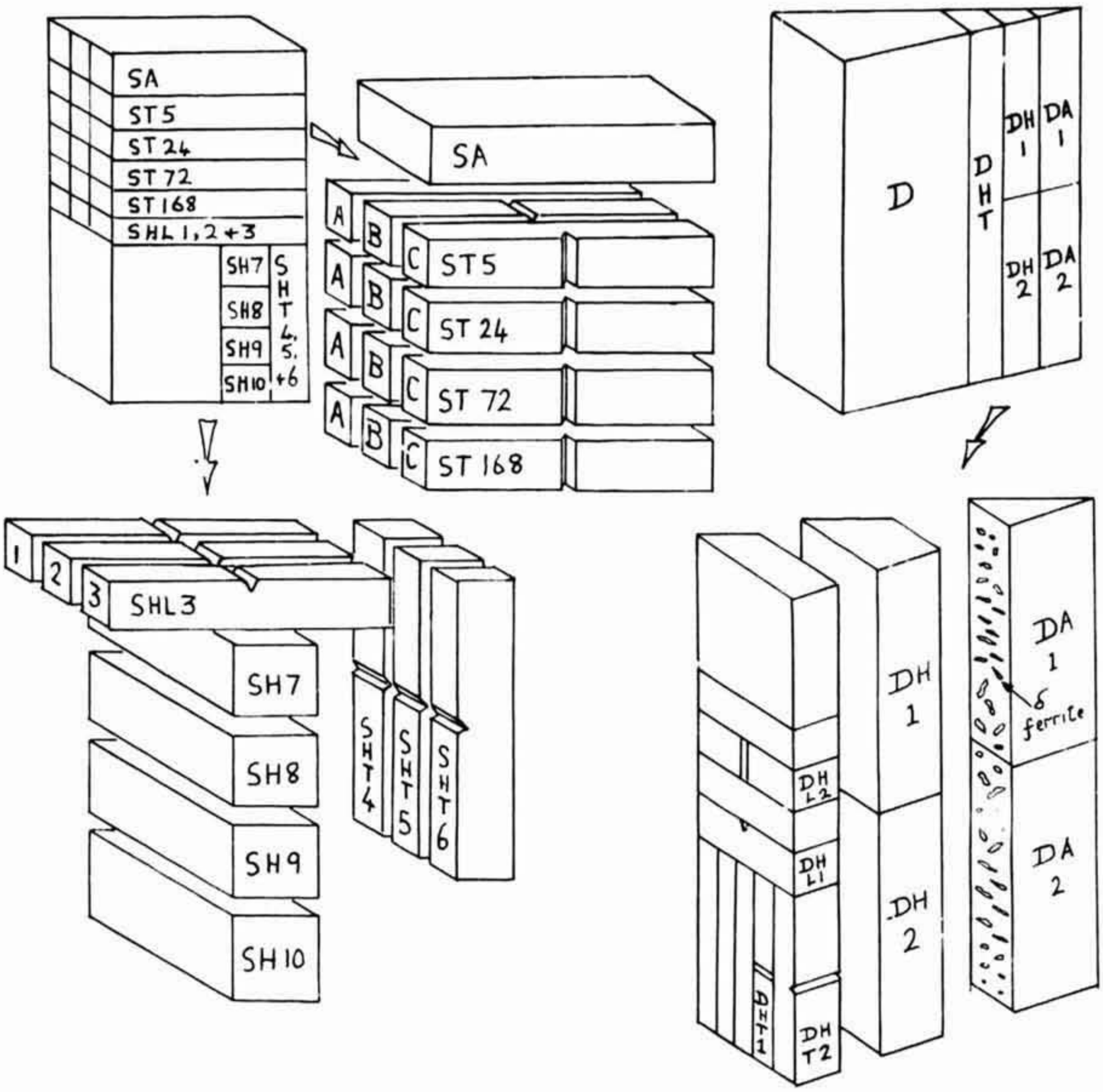


FIG.12: LOCATION OF SPECIMENS IN SAMPLES S and D FROM 5.5in. PLATE.

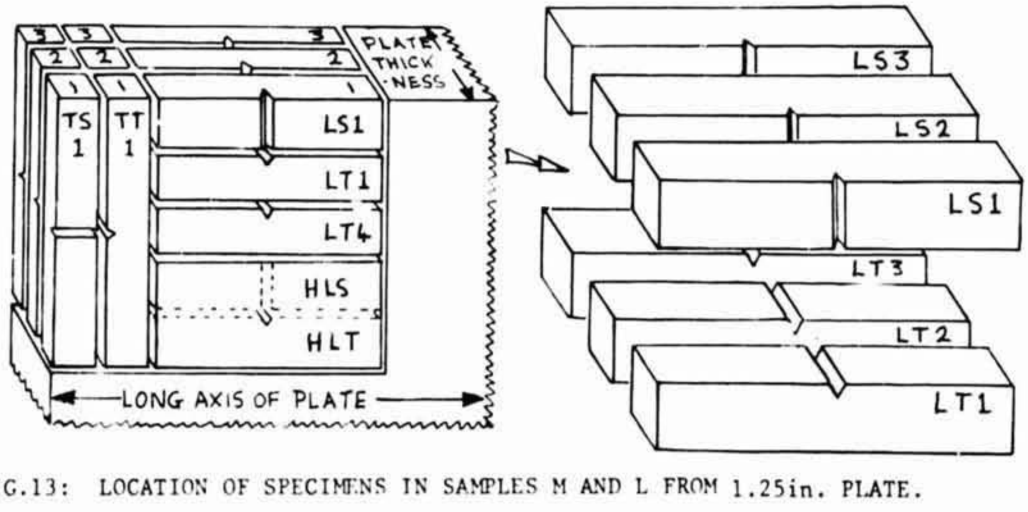


FIG.13: LOCATION OF SPECIMENS IN SAMPLES M AND L FROM 1.25in. PLATE.

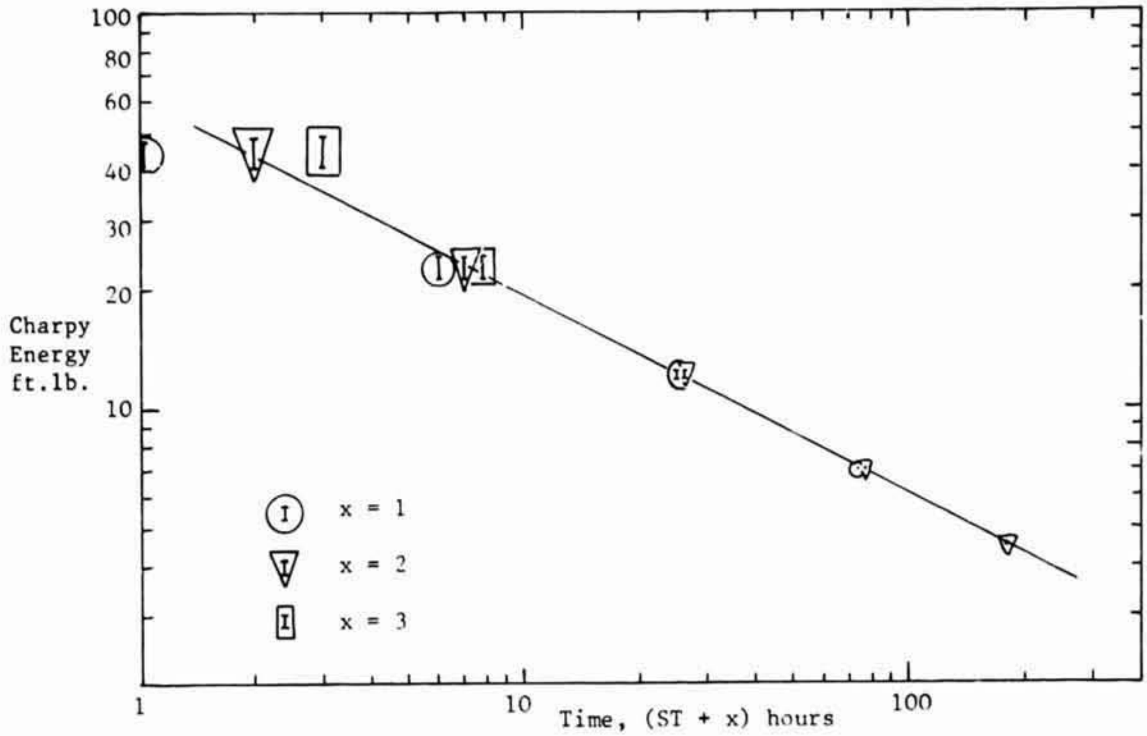


FIG. 14: RELATIONSHIP BETWEEN CHARPY ENERGY AND SENSITIZING TIME IN N40

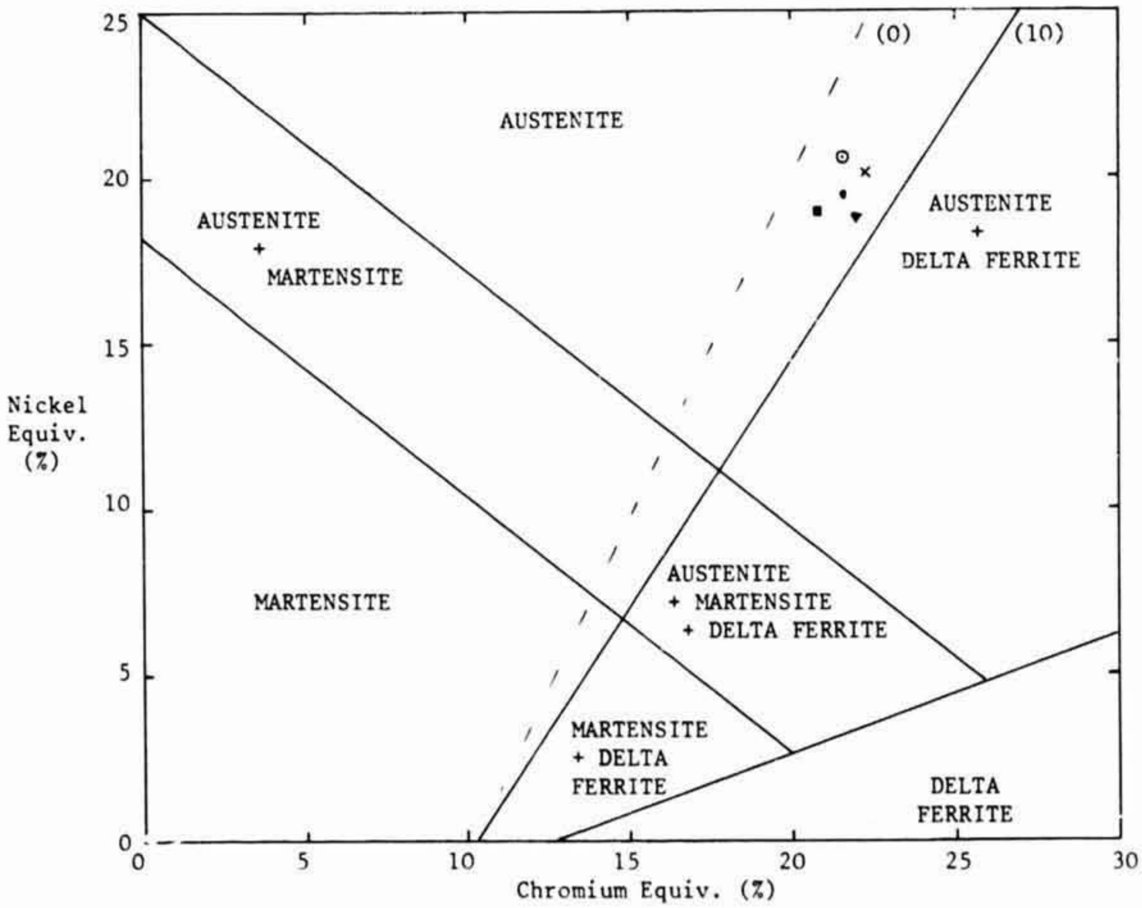
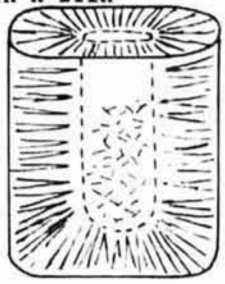


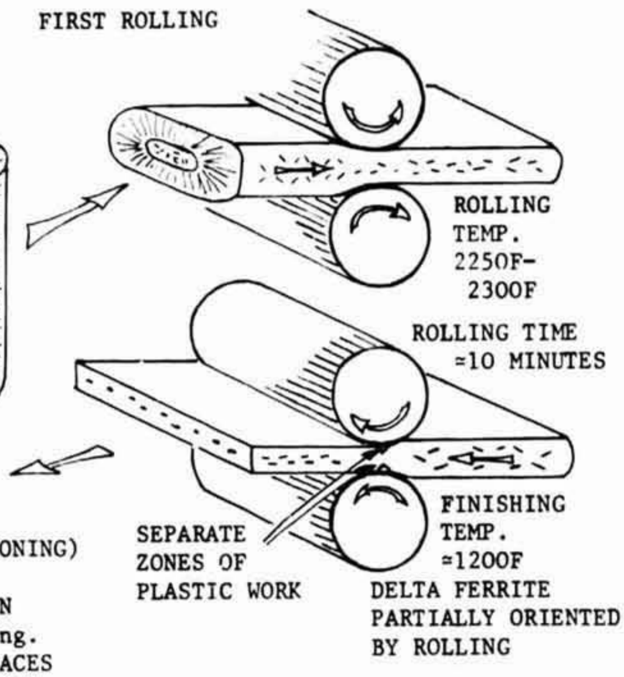
FIG. 15: SCHAEFFLER DIAGRAM SHOWING LOCATION OF NITRONIC 40 SAMPLES.

ARMCO/G.O. CARLSON
HEAT No.69310.

CAST INTO BILLET 52in x 21in

DENDRITIC GRAIN GROWTH FROM SURFACE CAUSES SEGREGATION OF ALLOYING ELEMENTS AND CONCENTRATION OF DELTA FERRITE WITH GRAIN BOUNDARY MORPHOLOGY AT CENTRE OF BILLET.



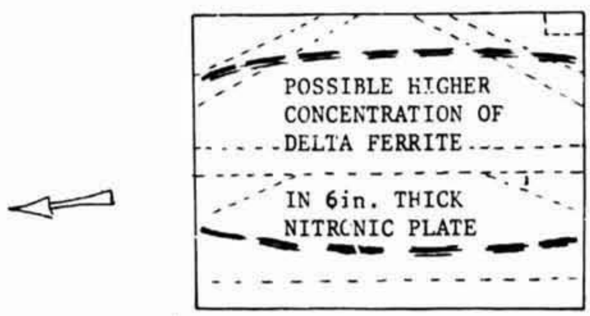


BILLET SIZE NOW
12in thick x 50in wide x 50in long
SURFACE DEFECTS GROUND OUT (CONDITIONING)

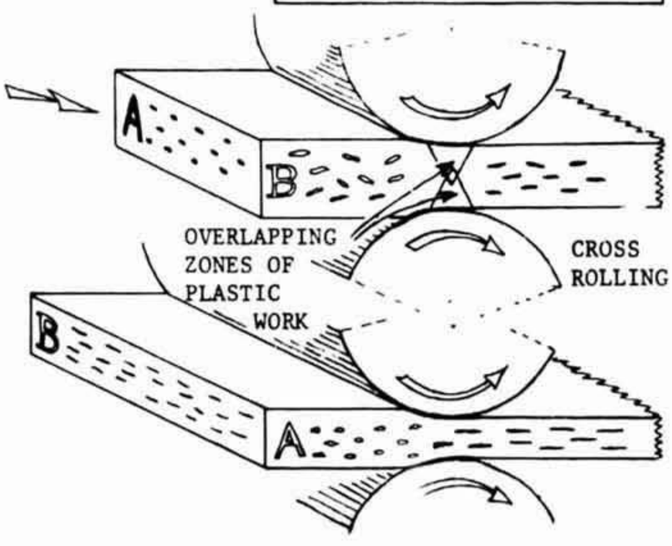
REHEATED TO 2275F FOR REROLLING DOWN TO 6in thick x 50in wide x 100in long. ENDS AND SIDES CUT OFF, ROLLED SURFACES GROUND TO GIVE SLAB 5.5in x 44in x 60in - DESIGNATED 69310-1F.

ZONES OF PLASTIC WORK DO NOT REACH CENTRE OF THICK PLATE: DELTA FERRITE LESS ORIENTED AT PLATE CENTRE

AS DELTA FERRITE ORIGINALLY IN MIDDLE OF BILLET, SIDES OF ROLLED SLAB PROBABLY CONTAIN LESS DELTA FERRITE THAN CENTRE, i.e. VARIATION OVER AREA OF PLATE



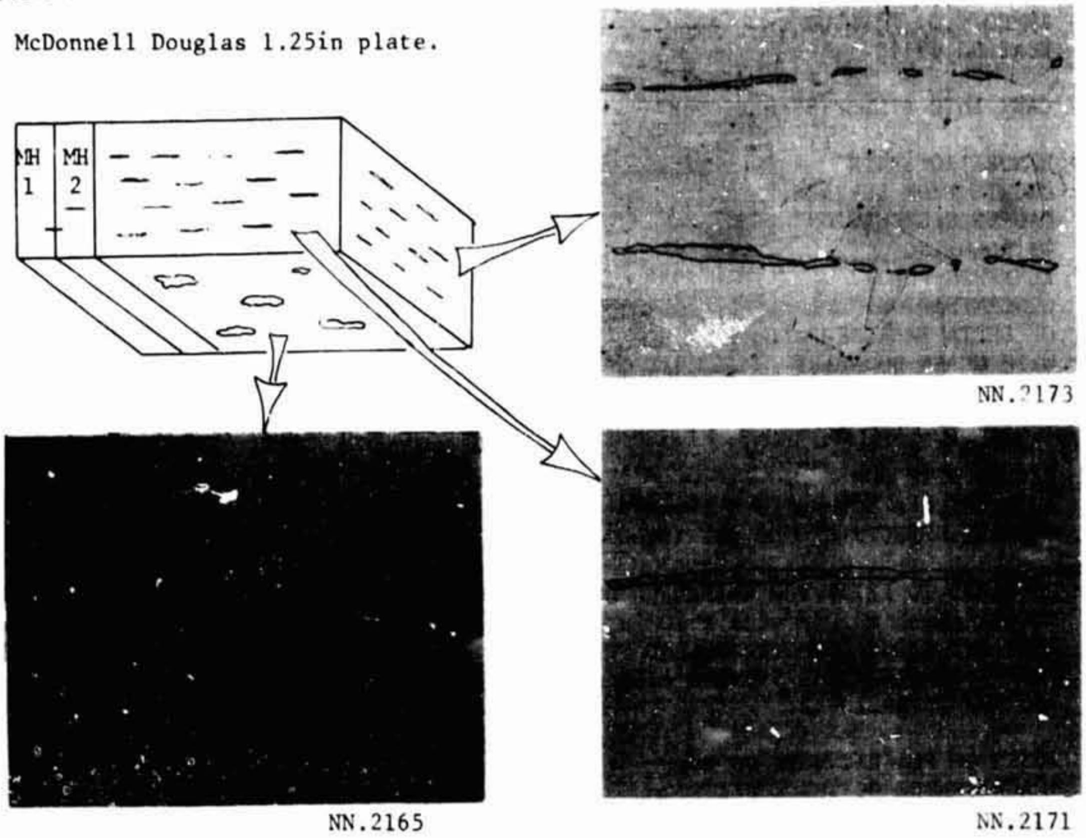
REMAINING MATERIAL FROM HEAT 69310 CROSS-ROLLED DOWN FROM 12in THICK.
2in thick x 40in x 130in
LANGLEY PLATE
DESIGNATED 69310-IE.
MCDONNEL DOUGLAS
1.25in x 13in x 52in PLATE
AND LOCKHEED 8 PLATES
1.25in x 14in x 60in
DESIGNATED 69310-1C.



CROSS ROLLING SPREADS DELTA FERRITE INTO DISCS WITH DIAMETER:THICKNESS RATIOS OF 10-20:1 AND SIMILAR DIAMETERS IN BOTH ROLLING DIRECTIONS.

FIG.16. SCHEMATIC REPRESENTATION OF THE EFFECT OF PROCESSING ON THE MORPHOLOGY OF DELTA FERRITE IN NITRONIC-40.

McDonnell Douglas 1.25in plate.



Pathfinder 1. Wing offcut

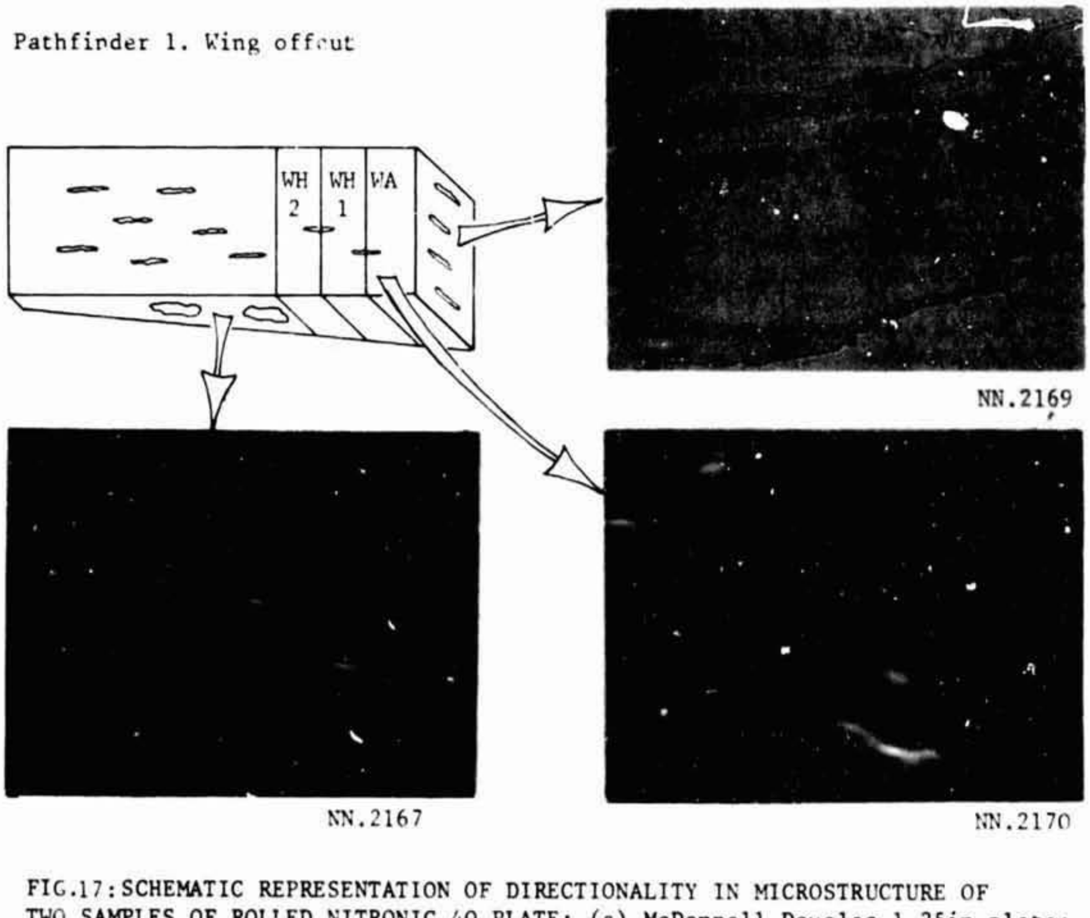


FIG.17: SCHEMATIC REPRESENTATION OF DIRECTIONALITY IN MICROSTRUCTURE OF TWO SAMPLES OF ROLLED NITRONIC 40 PLATE: (a) McDonnell Douglas 1.25in plate; (b) Offcut from Langley 5.5in plate for Pathfinder wing.(All magnif'n x 300).

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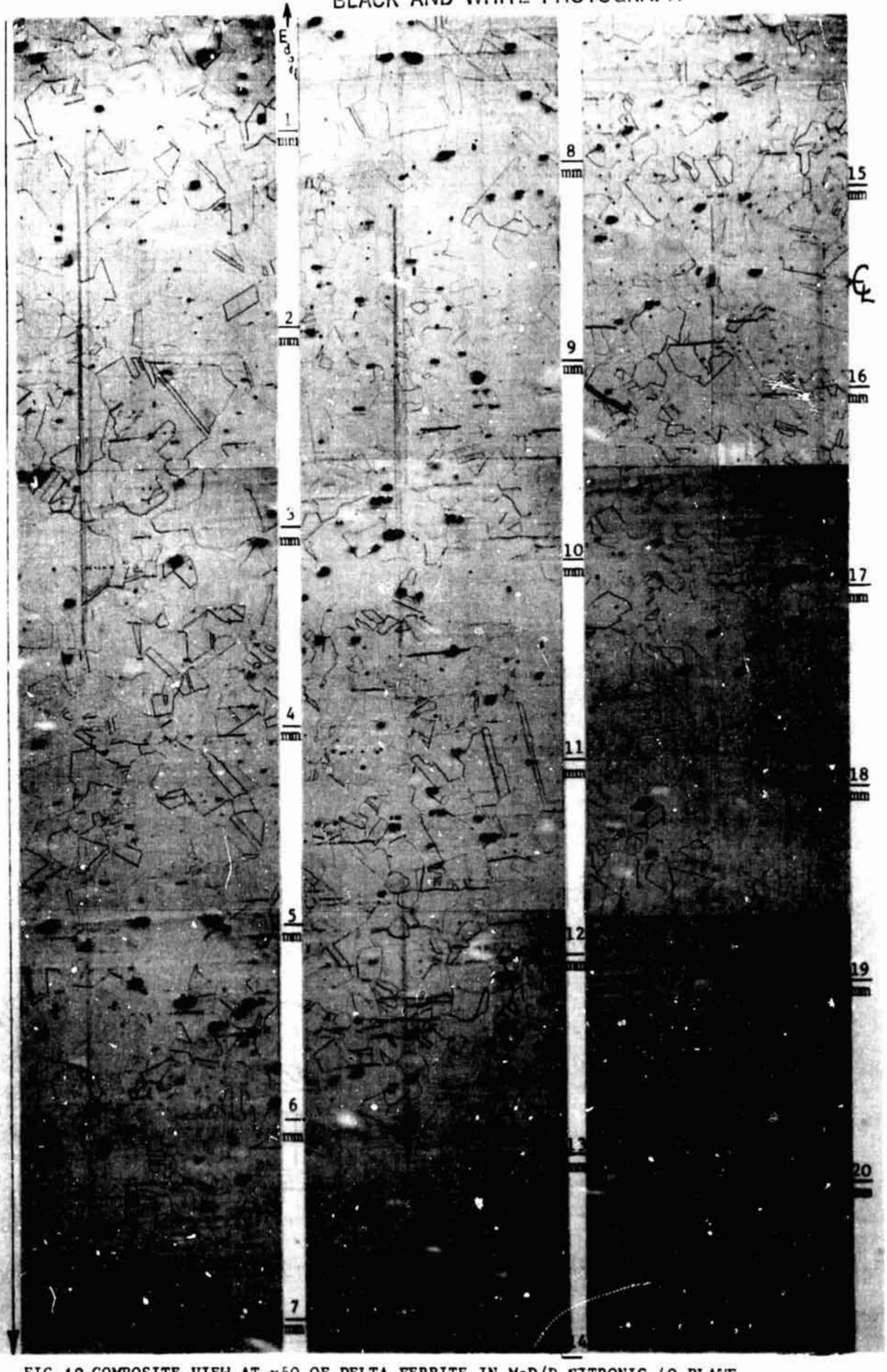


FIG. 18. COMPOSITE VIEW AT x50 OF DELTA FERRITE IN McD/D NITRONIC 40 PLATE

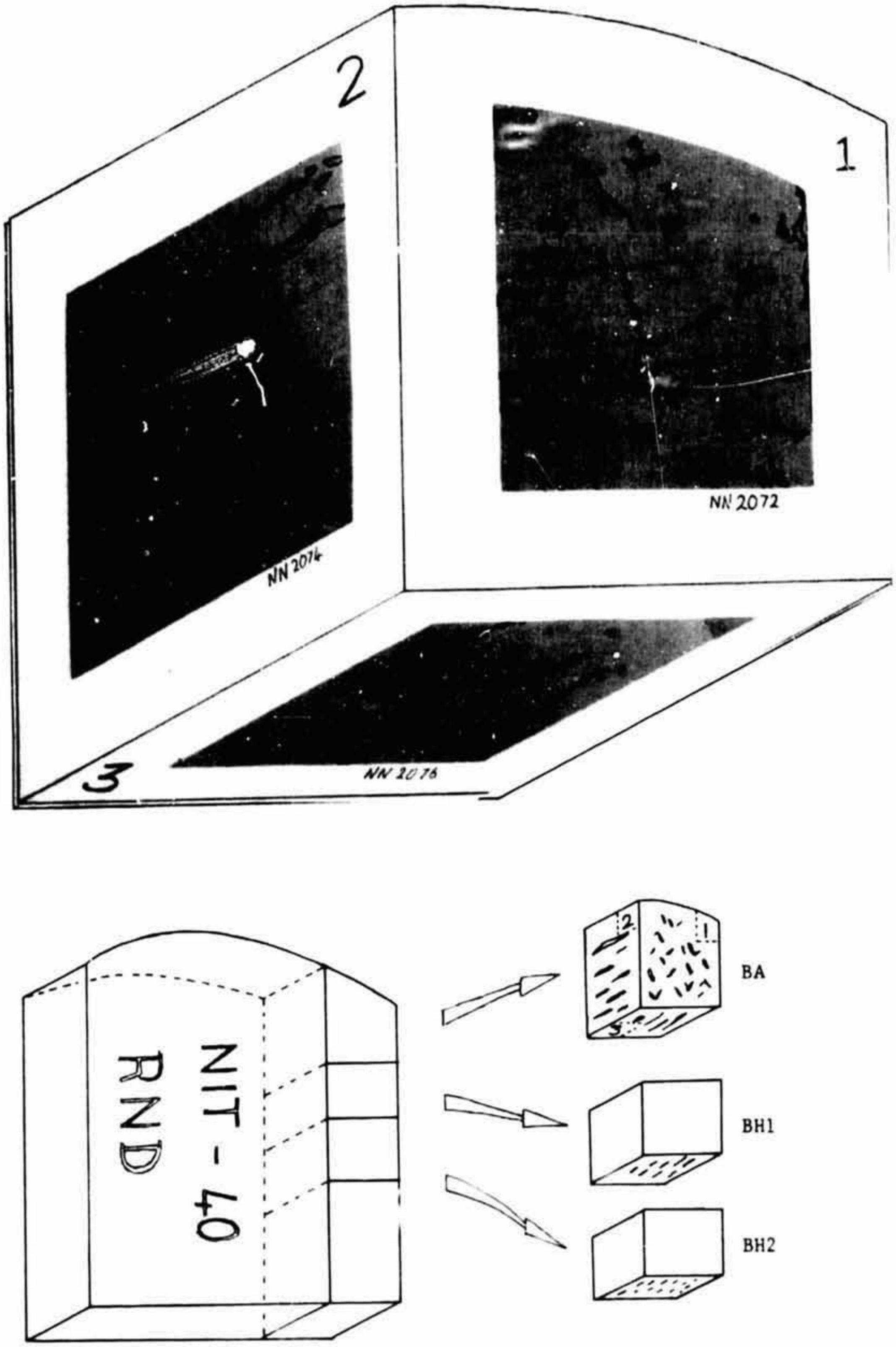
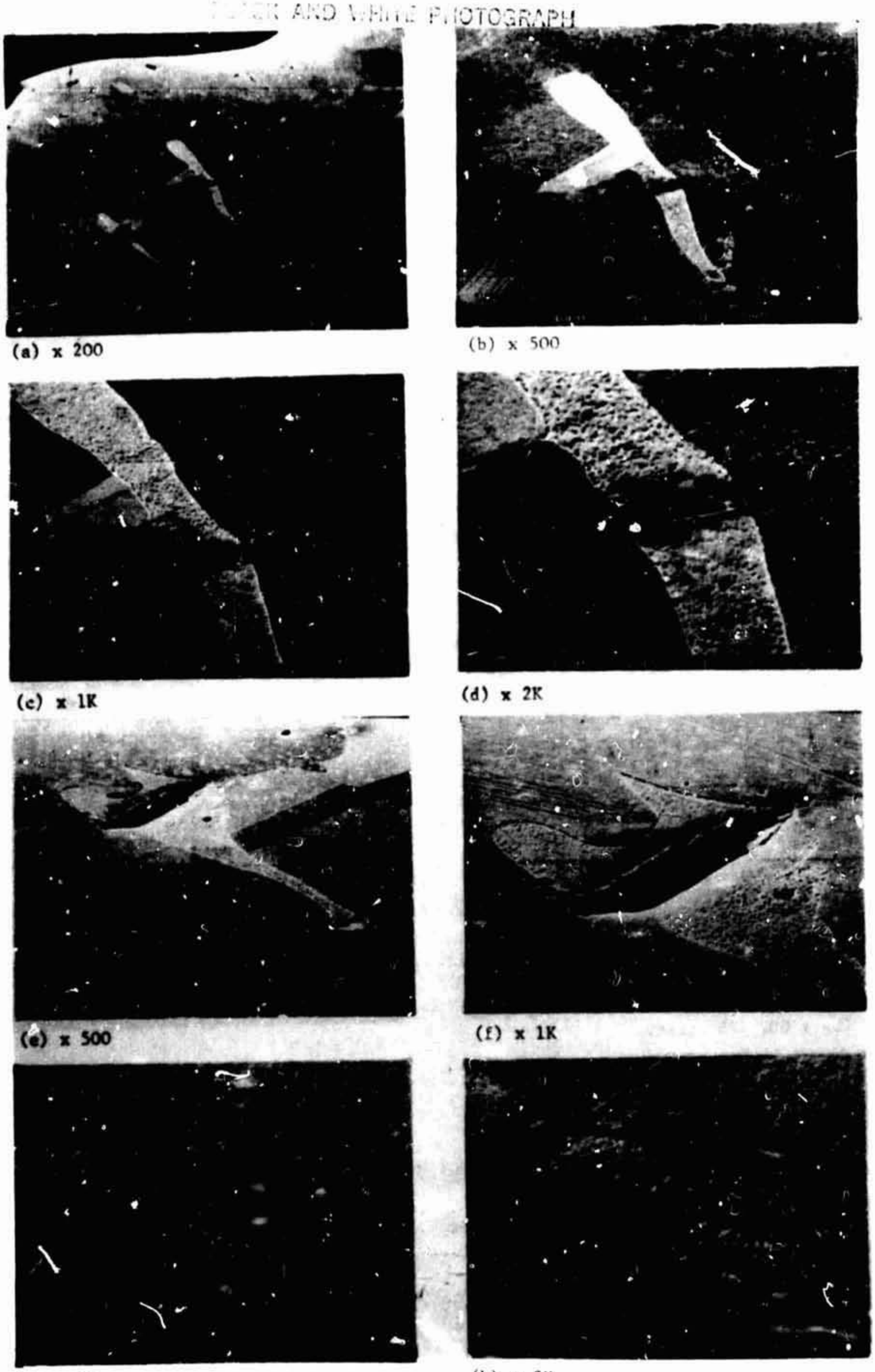


FIG. 19. SCHEMATIC REPRESENTATION OF DIRECTIONALITY IN MICROSTRUCTURE AND LOCATION OF SPECIMENS IN SAMPLE OF NITRONIC 40 ROUND BAR



(a) x 200 (b) x 500

(c) x 1K (d) x 2K

(e) x 500 (f) x 1K

(g) x 2K (h) x 2K

FIG.20: STEREOSCAN VIEWS OF CLEAVAGE CRACKS IN DELTA FERRITE ON POLISHED AND ETCHED SURFACE ADJACENT TO FRACTURE (Roll No.2144).

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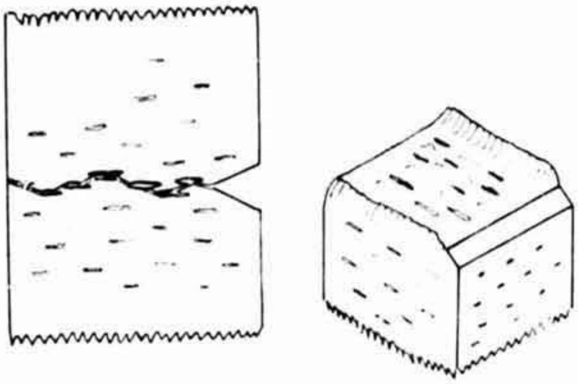
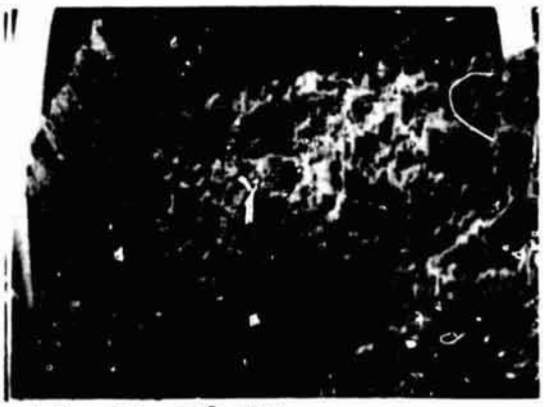
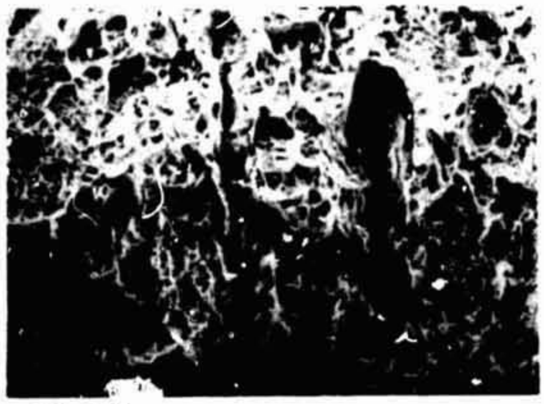


Schematic view of SHT4

(d) x 40, no tilt

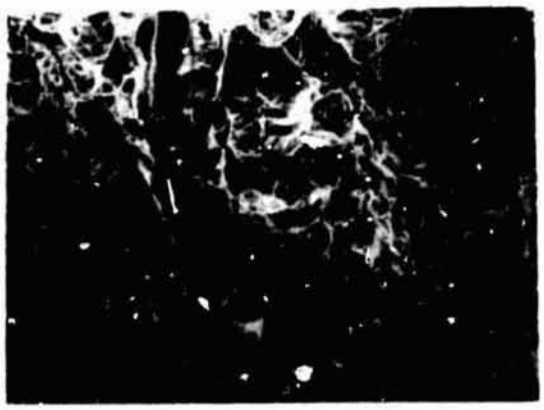


(a) x 13, 75° tilt.

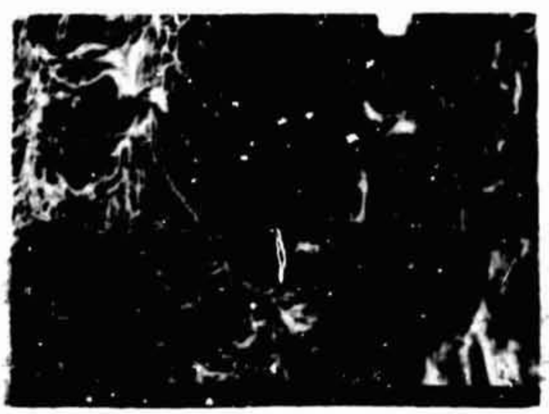


(e) x 80

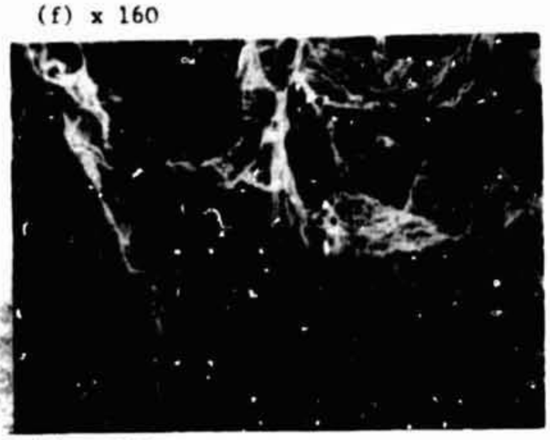
(b) x 63, 75° tilt.



(f) x 160

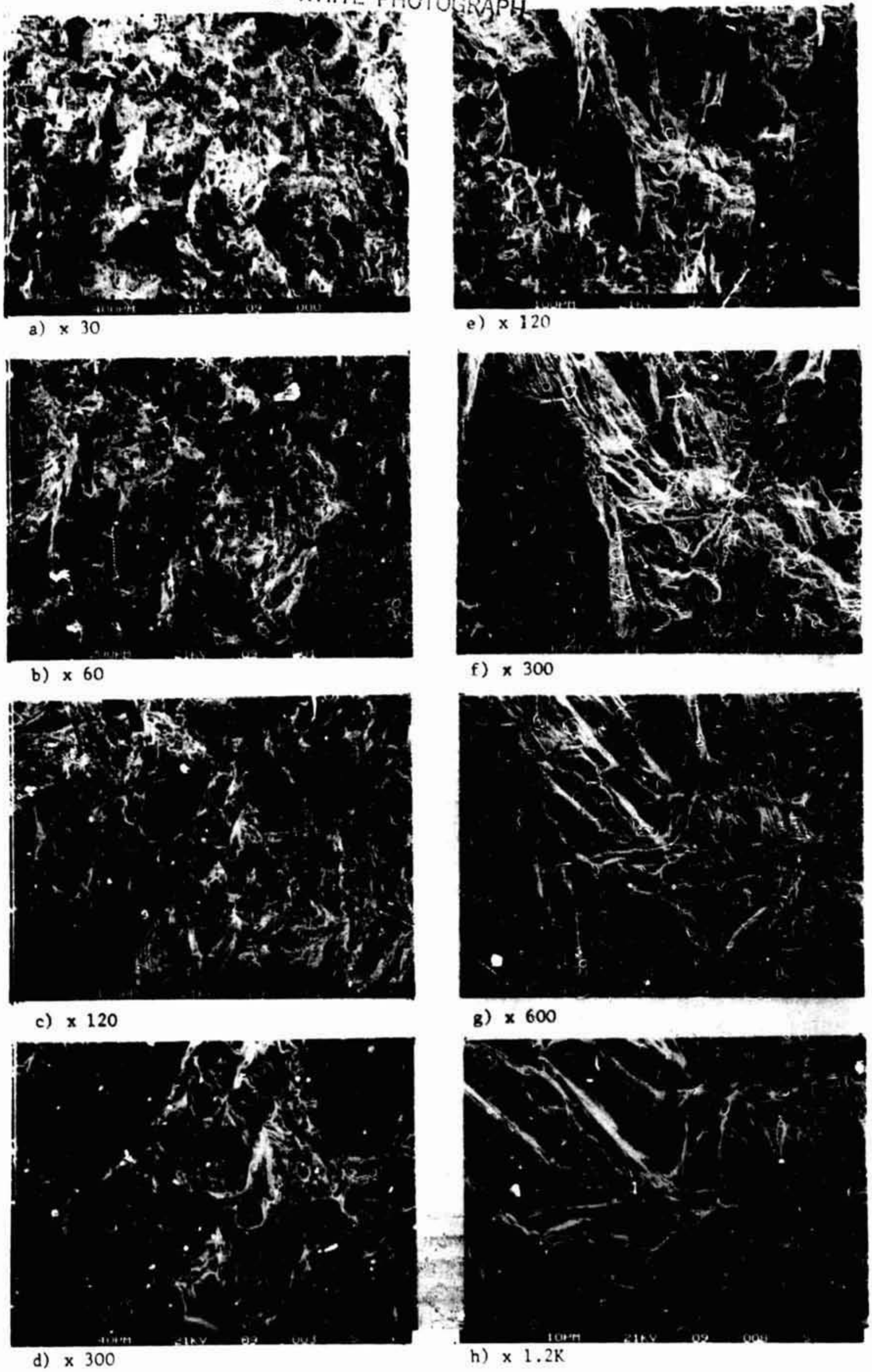


(c) x 300, 75° tilt



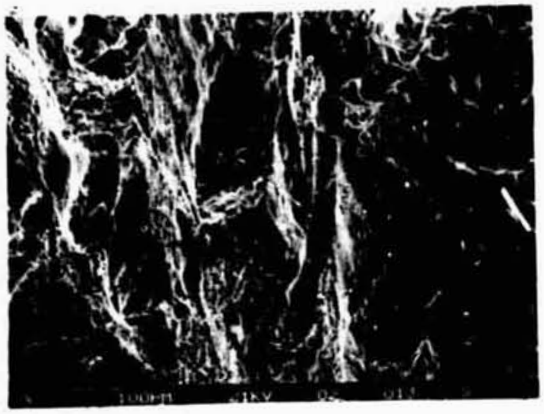
(g) x 400

FIG.21: STEREOSCAN VIEWS OF 7/8 CHARPY FRACTURE SURFACE OF DESENSITIZED N-40 WITH δ FERRITE ORIENTED PERPENDICULAR TO BAR (Roll Nos.2038 and 2180).

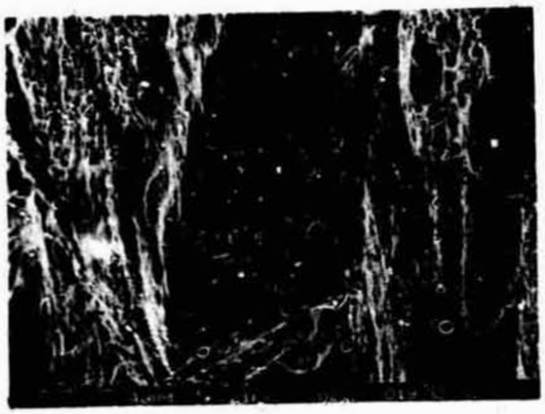


a) x 30
b) x 60
c) x 120
d) x 300
e) x 120
f) x 300
g) x 600
h) x 1.2K

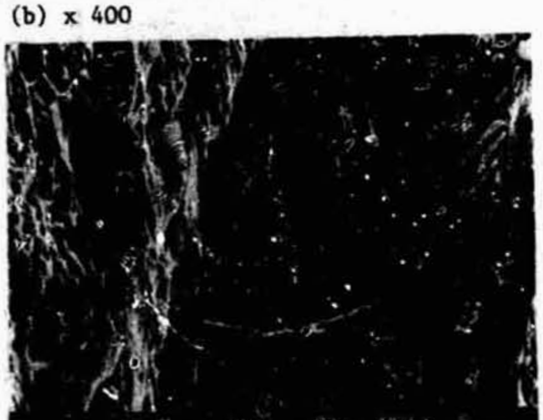
FIG.22: STEREOSCAN VIEWS OF -320F CHARPY FRACTURE SURFACE OF NITRONIC 40 SAMPLE SCVL9 SENSITIZED BY LANGLEY H.T. (Roll No.2207).



(a) x 160

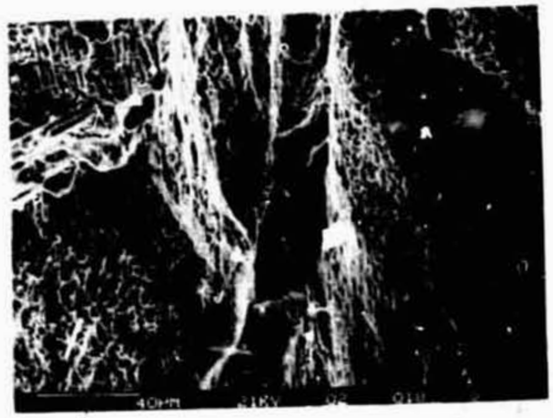


(b) x 400

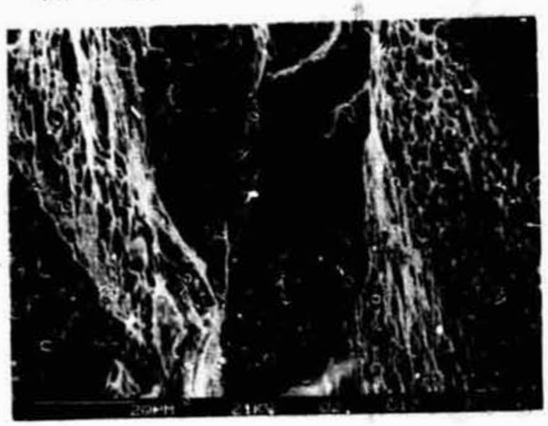


(c) x 800

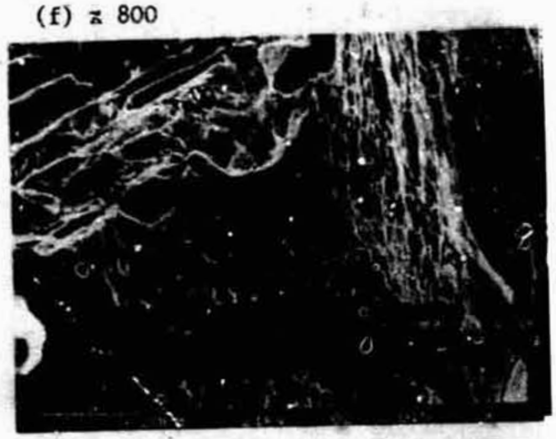
(d) x 1.6K



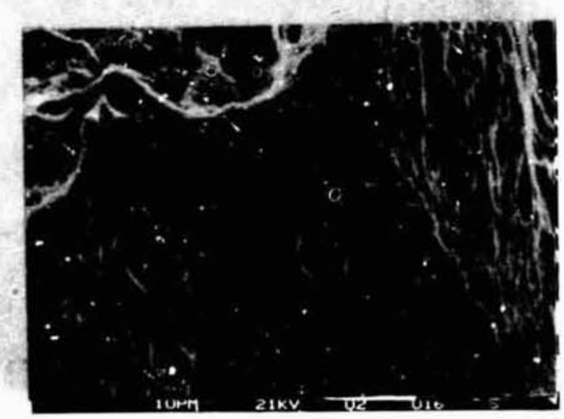
(e) x 400



(f) x 800

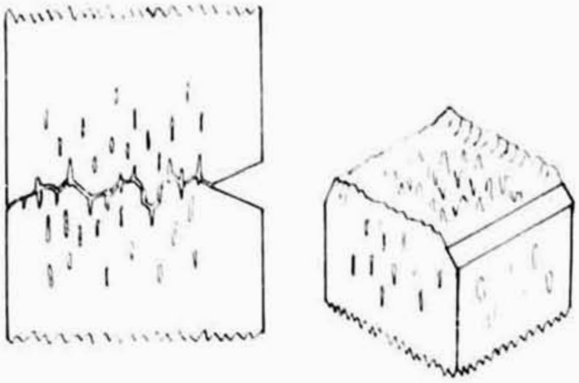


(g) x 800

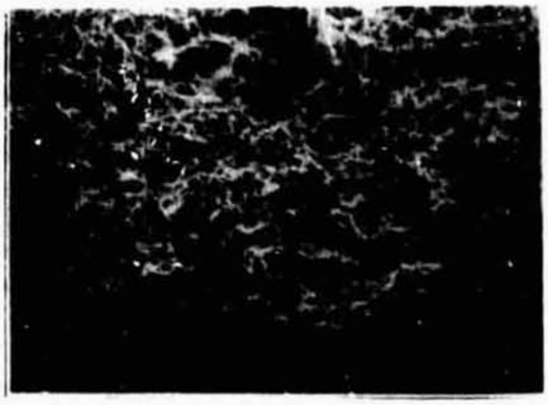


(h) x 1.6K

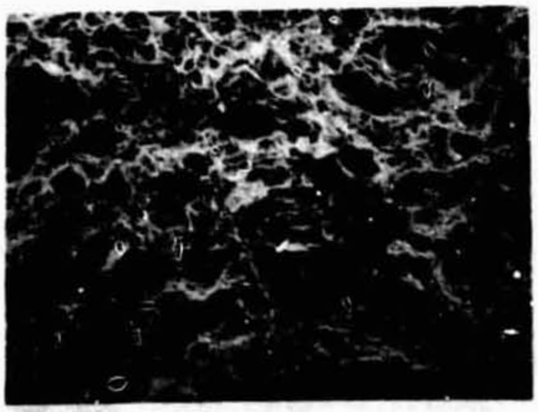
FIG.23: STEREOSCAN VIEWS OF -275F CHARPY FRACTURE SURFACES OF SENSITIZED N-40 (SCVL4) δ FERRITE ORIENTED PERPENDICULAR TO BAR) (Roll No.2180)



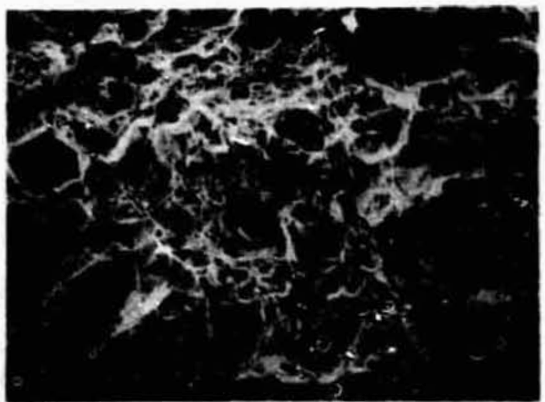
Schematic View of SHL1



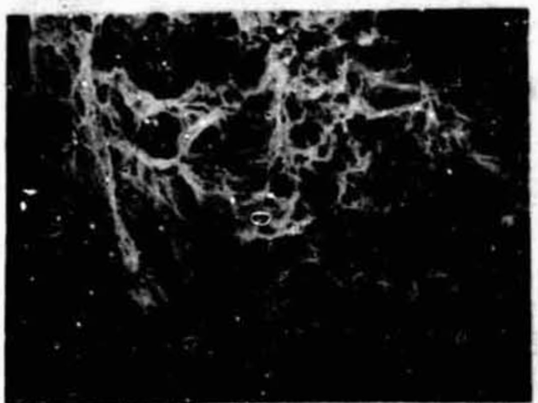
(a) x 16



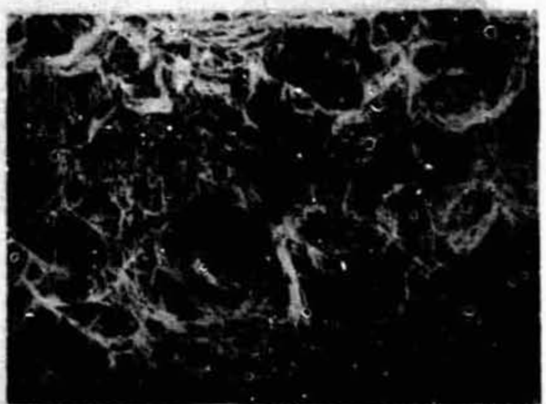
(b) x 40



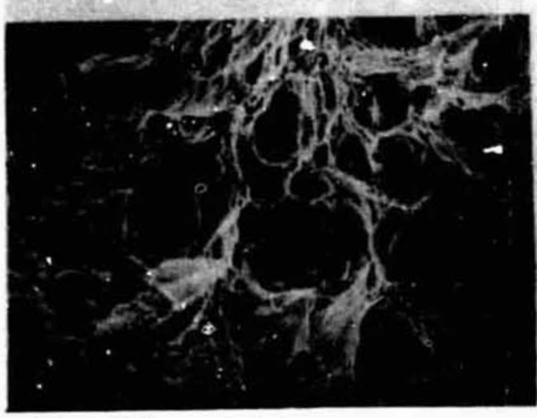
(c) x 80



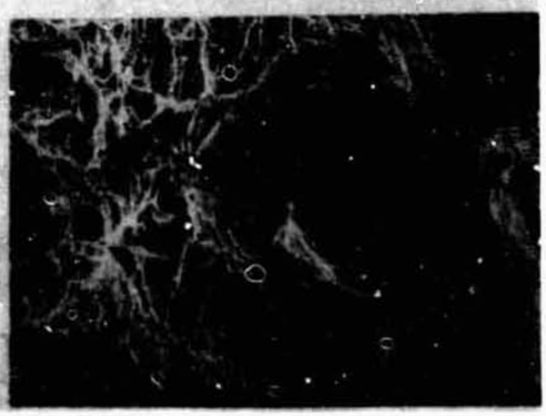
(f) x 80



(d) x 160

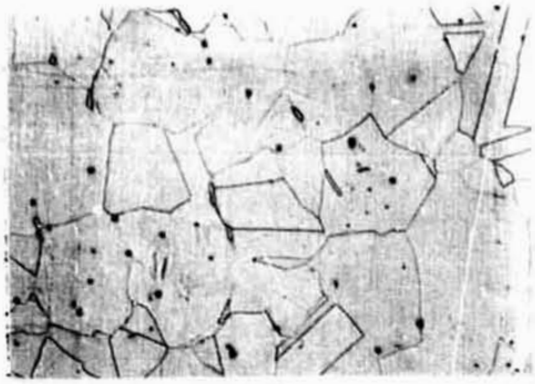


(g) x 400



(e) x 400

FIG.24: STEREOSCAN VIEWS OF 77K CHARPY FRACTURE SURFACES OF DESENSITIZED N-40 WITH δ FERRITE ORIENTED PARALLEL TO BAR (Roll No.2038)



a) SA x100, NN.2112

b) SA x300, NN.2113.

c) SH8 x100, NN.2050.

d) SH8 x300, NN.2051.

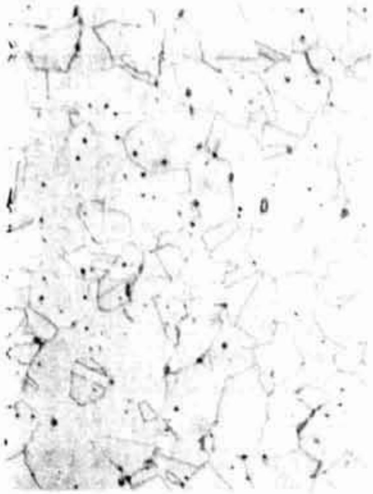
e) SH9 x100, NN.2048.

f) SH9 x300, NN.2049.

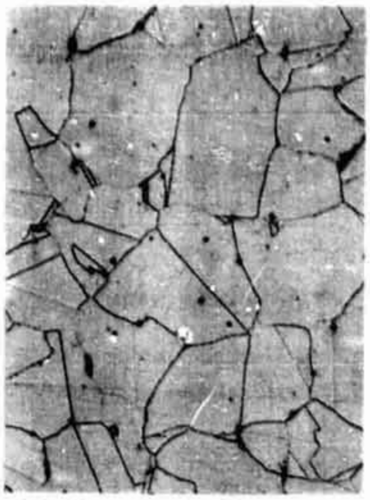
g) SH10 x100, NN.2046.

h) SH10 x300, NN.2047.

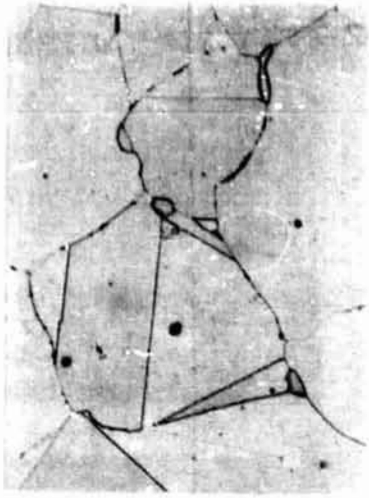
FIG.25: MODIFICATIONS TO DELTA FERRITE MORPHOLOGY AS A RESULT OF INITIAL HEAT TREATMENTS AT SOUTHAMPTON.
SA ≡ as received; SH8 ≡ 4hrs @ 1830F,1000C; SH9 ≡ 4.5hrs @ 2190F,1200C;
SH10 ≡ 0.5hr @ 2000F,1100C + 0.5hr @ 2190F,1200C + 1hr @ 2370F,1300C.



DA x50, NN.2108



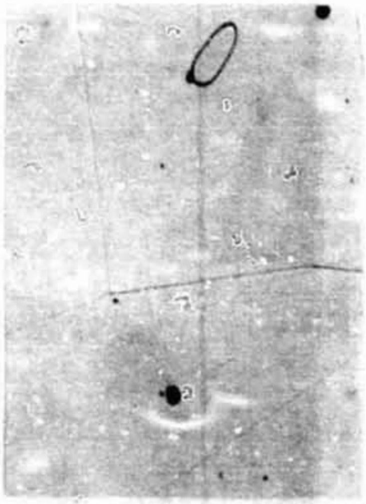
DA x100, NN.2109



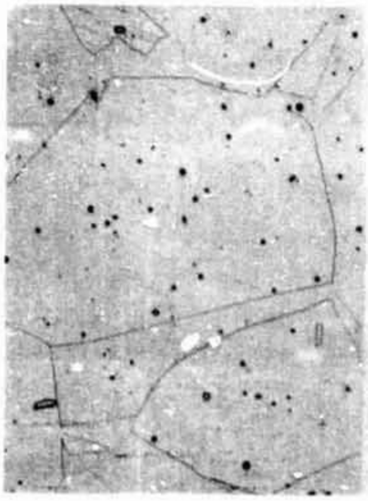
DA x300, NN.2110

DH1 x50, NN.2114

DH1 x100, NN.2115



DH1 x300, NN.2116



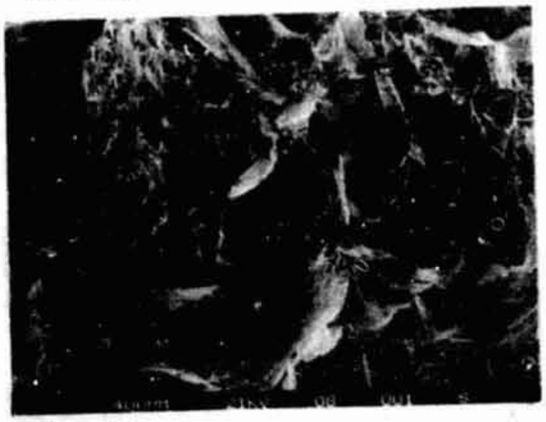
DH2 x50, NN.2117

DH2 x100, NN.2118

DH2 x300, NN.2119

FIG. 26. SAMPLE D: A (As received), H1 (2200F, 2hrs), H2 (2200F, 8hrs)

a) x 12.

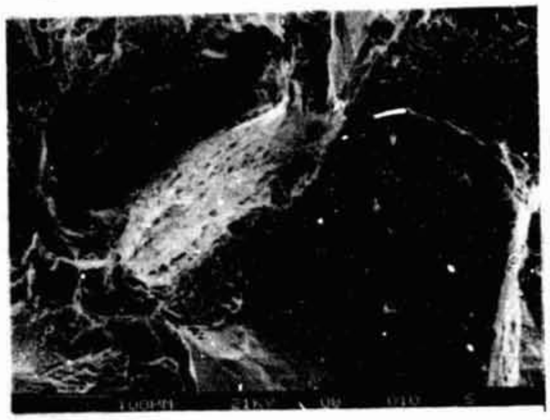


b) x 30

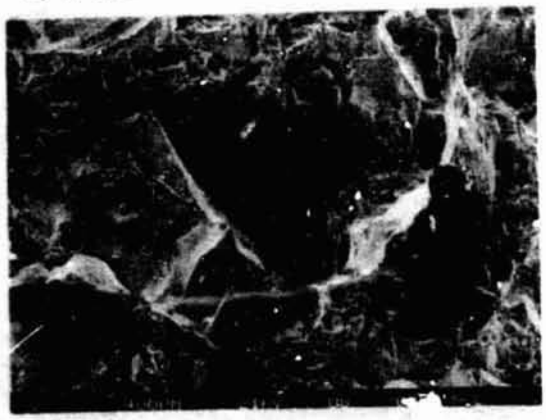
c) x 60

d) x 120

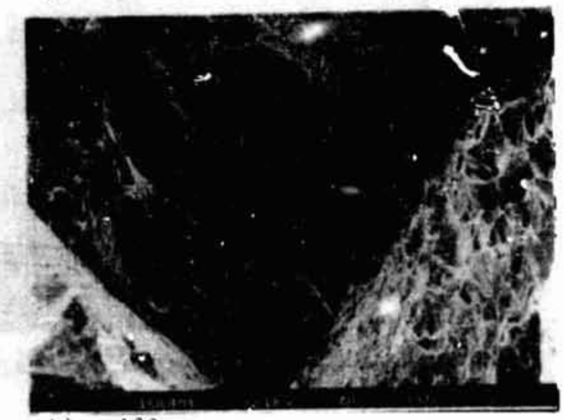
e) x 30.



f) x 120

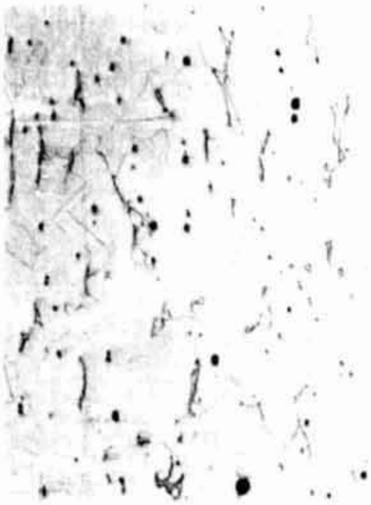


g) x 30

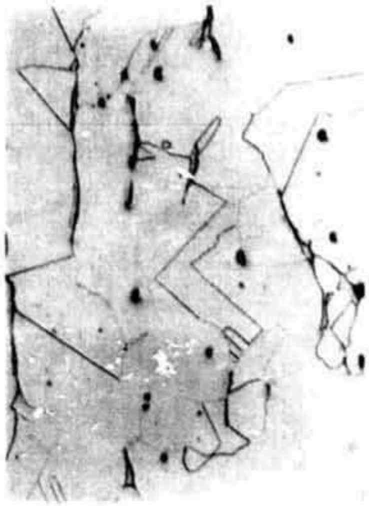


h) x 120

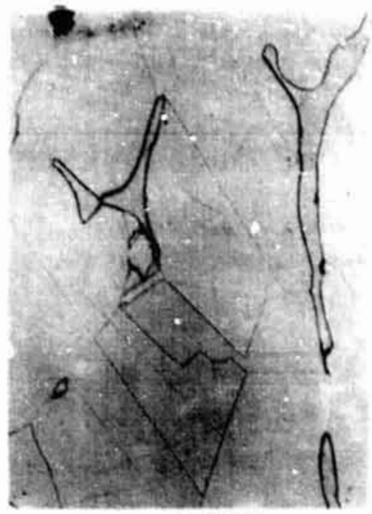
FIG.27: STEREOSCAN VIEWS OF -320F CHARPY SURFACE OF NITRONIC 40 SAMPLE DH2 SHOWING VERY LARGE GRAINS. (Roll No.2207).



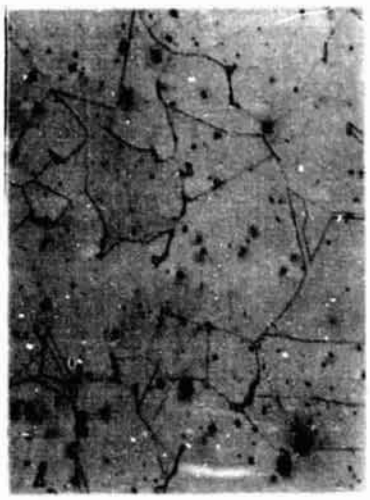
WA x50, NN.2081



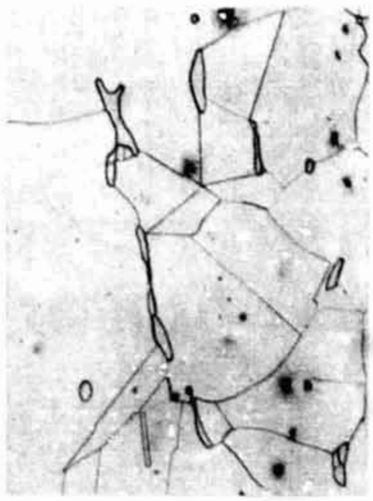
WA x100, NN.2082



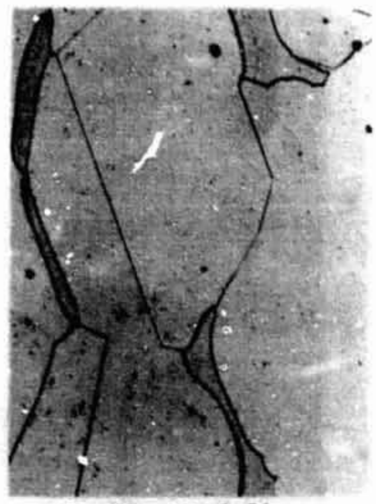
WA x300, NN.2083



WH1 x50, NN.2084

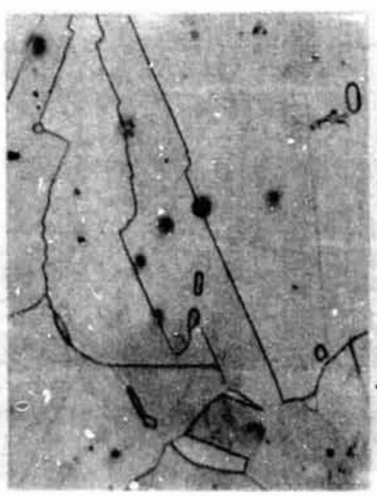


WH1 x100, NN.2085



WH1 x300, NN.2086

WH2 x50, NN.2087



WH2 x100, NN.2088

WH2 x300, NN.2089

FIG.28 SAMPLE W: A (as received), H1 (2200F, 2hrs), H2 (2200F, 8hrs)

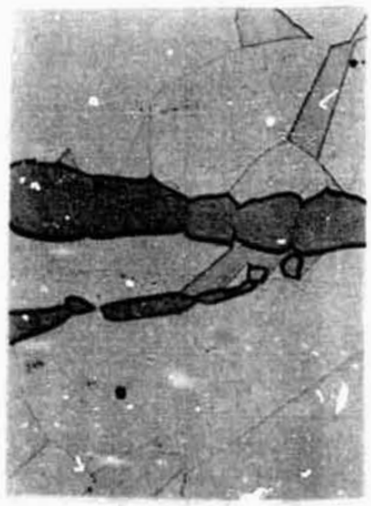
BA x50, NN.2090

BA x100, NN.2091

BA x300, NN.2092

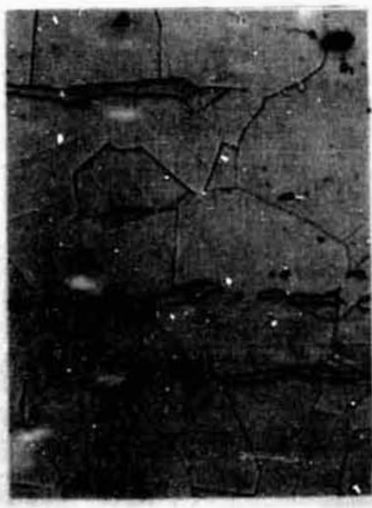
BH1 x50, NN.2093

BH1 x100, NN.2094

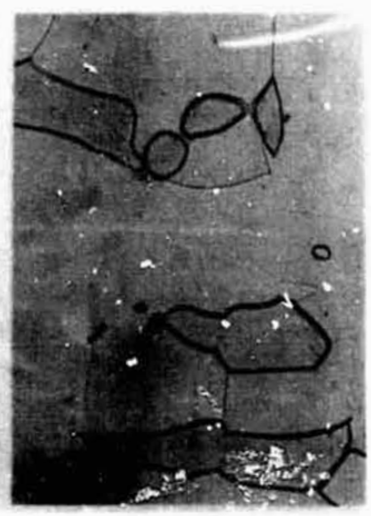


BH1 x300, NN.2095

BH2 x50, NN.2096



BH2 x100, NN.2097



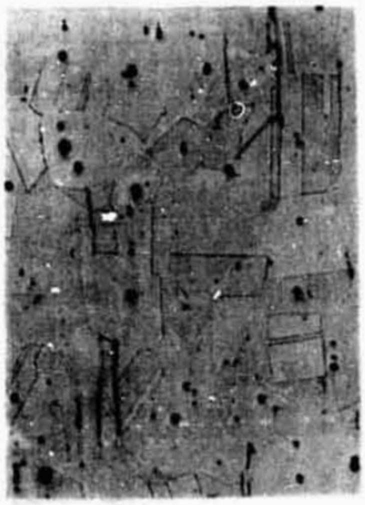
BH2 x300, NN.2098

FIG.29. SAMPLE B: A (As Received), H1 (2200F, 2hrs), H2 (2200F, 8hrs)

MA x50, NN.2099

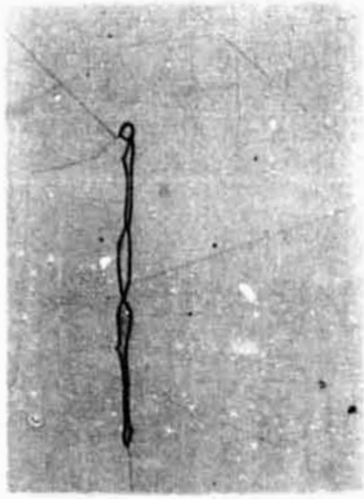
MA x100, NN.2100

MA x300, NN.2101

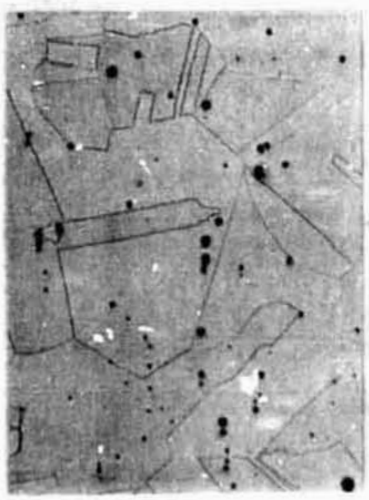


MH1 x50, NN.2102

MH1 x100, NN.2103

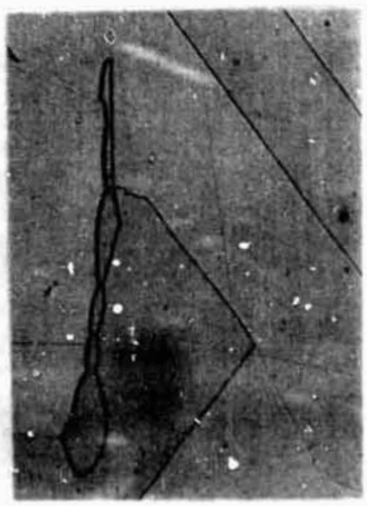


MH1 x300, NN.2104



MH2 x50, NN.2105

MH2 x100, NN.2106

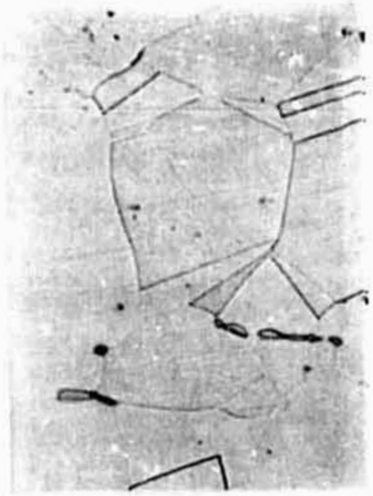


MH2 x300, NN.2107

FIG.30. SAMPLE M: A (As received), H1 (2200F, 2hrs), H2 (2200F, 8hrs)

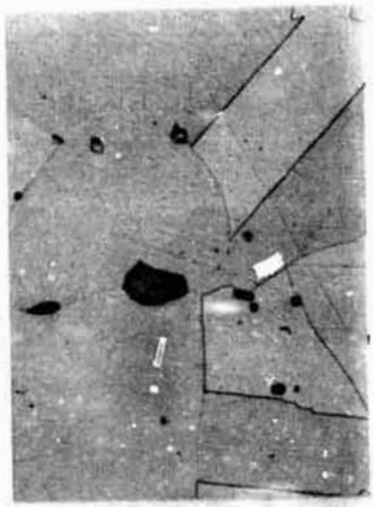

PA x50, NN.2120


PA x100, NN.2121


PA x300, NN.2122


PH1 x50, NN2123


PH1 x100, NN.2124


PH1 x300, NN.2125


PH2 x50, NN.2126


PH2 x100, NN.2127


PH2 x300, NN.2128

FIG.31. SAMPLE P: A (as received), H1 (2200F, 2hrs), H2 (2200F, 8hrs)