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DEVELOPMENT AND METALLURGY STUDY OF A NASA COBALT-BASE SUPERALLOY

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PHILCO-FORD CORPORATION
AERONUTRONIC DIVISION

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
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F. H. Harf, Project Manager

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FINAL REPORT

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ABSTRACT

VM-103, previously a Co-25W-3Cr-1Ti-0.5Zr-0.5C research laboratory superalloy, was further advanced by developing forging, hot rolling, and cold rolling parameters for fabrication of 25-50 lb. (11-23 kg) ingots produced by induction plus vacuum arc remelting and induction plus electroslag remelting. Electroslag remelted VM-103 proved superior in respect to strength, ductility, and fabricability. Aging studies showed significant hardening effects on prior annealed and prior cold-worked material. The 2200°F (1205°C) yield strength was increased by 140% by aging prior annealed material. The fabrication studies, conventional and high strain rate tensile tests, fatigue tests, and bend tests indicated that VAR-103 is competitive with conventional superalloys, particularly for short time high temperature applications.

1. SUMMARY

The objective of this program was to advance VM-103, a Co-25W-3Cr-1Ti-0.5Zr-0.5C superalloy from a research laboratory status to the level of an advanced superalloy, usable for numerous high temperature applications.

To accomplish this objective, a fabrication development and physical metallurgy study was conducted on five 25-50 lb. (11-23 kg) ingots, two produced by induction plus vacuum arc remelting and three produced by induction plus electroslag remelting. Processing parameters for primary and secondary fabrication were developed, and 0.012 in. (0.30 mm) thick foil was produced from the 4 in. (10 cm) diameter ingots. The processes included hammer forging, hot rolling, and cold rolling. This work showed that VM-103 can be produced and fabricated by production oriented processes and is relatively fabricable. The electroslag remelted ingots showed significantly higher fabricability than the vacuum arc remelted material.

Mechanical testing consisting of conventional and high strain rate tensile tests, bend tests, and limited fatigue tests was conducted to establish properties of wrought material and ascertain differences between results of the two basic melting processes. Conventional tensile tests at 75 and 1600-2200°F (24°C and 870-1205°C) showed properties equal to or better than the early NASA laboratory heats. High strain rate tests (at 5/minute) showed the alloy to be very strain-rate sensitive and also indicated that strengthening effects of cold work were retained for short times at 1800°F (980°C). The limited bend and fatigue tests indicated superiority of the electroslag remelted material; this agreed with the tensile results which showed generally higher strengths and ductilities on electroslag vs. vacuum arc remelted material.

Aging studies were conducted on prior annealed and prior cold-worked material to investigate possible strengthening mechanisms. Aging treatments from 700-1600°F (370-870°C) for 1-100 hours were found to be effective in hardening prior cold-worked material, and to a lesser degree, prior annealed material. Tensile tests at 2200°F (1205°C) on prior annealed and aged samples showed an increase in yield strength of approximately 140%. More detailed study of this phenomenon is required.

Based on the results of the program, it was concluded that VM-103 is a producible, fabricable, high strength alloy which is competitive with other conventional nickel and cobalt base superalloys, particularly for short time high temperature applications. Further work in areas of compositional control and thermomechanical processing is recommended.

2. INTRODUCTION

This report summarizes the results of a NASA-funded program with the objective of further developing VM-103, a NASA high strength cobalt base superalloy. This alloy, with a nominal composition of Co-25W-3Cr-1Ti-0.5Zr-0.5C, shows potential for various high temperature applications due to its excellent high temperature strength properties. It also appears to be competitive with conventional superalloys such as L-605, René 41, Hastelloy X, and Waspaloy for numerous aerospace and ordnance high temperature applications.

NASA's early research work on cobalt-tungsten alloys, conducted by Freche, et al.,[1-4] involved systematic alloying studies wherein various compositions were evaluated primarily with respect to elevated temperature properties and fabricability. This work was conducted on vacuum or inert atmosphere single induction melted heats of 3-4 lb. (<2 kg). The VM-103 composition appeared to be very promising.

Subsequently, in seeking improved superalloys for various in-house design requirements, Aeronutronic conducted an internally funded effort to generate more complete information on VM-103 regarding mechanical properties, fabricability, weldability, compositional effects, and applicable melting processes. During Aeronutronic's program, various hardware items related to missile hot gas valves and high cyclic rate gun components were fabricated from VM-103 and successfully tested. In order to accomplish this work, 25-50 lb. (11-23kg) ingots of VM-103 were successfully produced by two production oriented duplex melting processes, i.e., the conventional vacuum induction + vacuum arc remelt (VAR) and the relatively new induction + electroslag remelt (ESR) processes.

The program discussed in this report followed at Aeronutronic under NASA funding. The program initiated in March 1969 was designed to further advance VM-103 technology by conducting fabrication processes development and physical and mechanical metallurgy studies. Hot and cold working parameters and thermal treatments for processing the alloy from ingot to bar, sheet, or foil were developed. A metallurgical study established the effects of melting and processing on mechanical properties and aided in improving an understanding of the basic physical metallurgy of the alloy.

The program was divided into five tasks, briefly described below.

(1) Task I - Hot Working Study

This task involved determining optimum hot working and annealing parameters for the alloy and establishing the effects of melting process (i.e., VAR versus ESR) on hot working characteristics.

(2) Task II - Cold Working Study

This task was designed to develop optimum cold working and annealing parameters for producing thin VM-103 sheet and foil and to determine the effects of the melting process on cold workability.

(3) Task III - Mechanical Property Evaluation

This task involved tensile and fatigue testing of annealed and cold worked alloy, and included a comparison of VAR versus ESR material properties.

(4) Task IV - Aging Recrystallization and Microstructure Study

This task was designed to determine effective aging treatments for the alloy and to recommend maximum short time service temperatures for cold worked material. A correlation of microstructure with processing variables and mechanical behavior resulted in an improved understanding of the physical metallurgy of the alloy.

(5) Task V - Evaluation of an Ingot with Improved Composition

This task was added during the program for purposes of evaluating a 50 lb. ESR heat with improved composition control with respect to fabricability and mechanical properties.

As discussed below, successful completion of these tasks resulted in encouraging data and in a significant advancement in knowledge of the alloy's properties.

3. PROCEDURE

Starting Material

As indicated above, most of the early work on VM-103 had been conducted on small 3-4 lb. (<2 kg) vacuum or inert atmosphere single induction melted heats. This process is very convenient and appropriate for research work but is not usually considered acceptable for wrought superalloy production due to inherent microsegregation and relatively high impurity levels. Duplex melting methods are frequently used such as the conventional vacuum induction + vacuum arc remelting (VAR), and more recently, the induction + electroslag remelting (ESR) processes. ESR is a relatively new process that has been shown to generally improve such properties as ductility, fabricability, fatigue strength, and fracture toughness of various steels and nickel base superalloys.[5,6]

Two 25 lb. (11 kg), 4 in. (10 cm) diameter VAR ingots melted at Aeronutronic, designated hereafter as 20-1 and 20-5, were selected for use on this program. In addition, 50 lb. (23 kg) ESR heats (melted from the same raw stock as the VAR ingots) designated PF-11 and PF-13 were supplied by ESCO Corporation, Portland, Oregon. Figure 1 shows the two original as-cast ESR heats and a typical VAR heat 20-1. Chemical analyses of the VM-103 heats are presented in Table I. All metallic elements reported were determined by X-ray spectroscopy with an estimated standard deviation of $\pm$ 25% for Ti and Zr, and $\pm$ 10% for the remaining elements. Carbon was determined by gas analysis with an estimated standard deviation of less than $\pm$ 2%.

Because the analyses of the initial four heats indicated a need for improved compositional control, Heat No. PF-288 was supplied by NASA (purchased from ESCO) near the end of the program. This heat, although an improvement in some respects, still did not meet the targets for tungsten and zirconium. Although the results of work performed on these five heats were very encouraging as discussed in Section 4, additional melting process development may result in improved properties and fabricability. Levels of alloying elements such as zirconium and titanium should be better controlled, and effects of impurities such as Fe, Ni, Mn, and Si should be better understood.

Forging

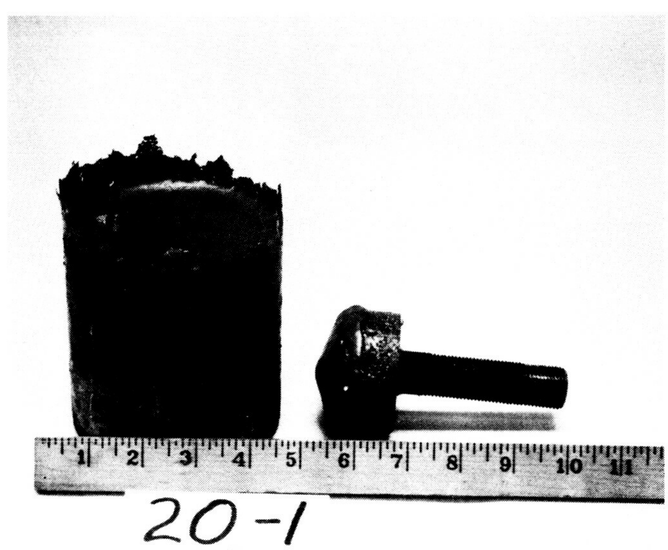
Prior to this program, the primary hot working of VM-103 was essentially limited to hot rolling small cast pieces. In order to more closely simulate superalloy production processes for producing billets from cast ingots, hammer forging was selected for working the 4 in. (10 cm) diameter ingots to 1 x 1 in. (2.5 x 2.5 cm) bar. Because hammer forging is usually considered

TABLE I

CHEMICAL ANALYSES OF VM-103 HEATS
(Weight Percent)

		VAR		ESR		
	Target Analysis	20-1	20-5	PF-11	PF-13	PF-288
W	25	26.89	26.71	24.13	23.40	28.15
Cr	3	2.56	2.63	2.51	2.83	3.00
Ti	1	0.94	1.45	0.95	1.47	1.06
Zr	0.5	0.89	0.73	0.25	0.30	0.24
C	0.5	0.57	0.55	0.50	0.49	0.45
Co	Balance	Bal.	Bal.	Bal.	Bal.	Bal.
Fe	0.1 Max.	0.18	0.12	1.08	1.64	0.16

As-Received ESR Ingots PF-11 and PF-13.



As-Cast VAR Heat 20-1 and Remainder of Consumable Electrode.

FIGURE 1. VM-103 INGOTS USED FOR THIS PROGRAM

to be a severe test of hot workability, particularly for a cast structure, this process was selected as a conservative assessment of VM-103 hot working characteristics. A total forging reduction of approximately 12:1 was selected for primary working in order to assure that maximum homogeneity and resulting properties could be achieved. Superalloys are commonly hot worked at least 8:1 prior to usage.

The ESR ingots were sectioned; one-half of each was set aside as backup material, while the other half was forged along with the entire VAR ingots. The forging was conducted at West Coast Forge, Compton, California, with a 3500 pound (15,500 N) hammer forge. Based on previous work at NASA and at Aeronutronic involving successful hot rolling of cast VM-103, a temperature range of 2150°-2200°F (1175-1205°C) was selected and used for forging trials on a small section of VAR heat 20-5. The preliminary forging trials led to the following procedure which was used successfully for the five ingots:

(1) Soak at 2175°F (1190°C) for 1/2 hour.

(2) Forge in radial direction to 3 in. x 3 in. (7.6 x 7.6 cm) square using reductions of approximately 8-10%.

(3) Return to furnace after each reduction; soak at temperature for 15 minutes.

(4) Forge to 1 in. x 1 in. (2.5 x 2.5 cm) square using 15-20% reductions.

(5) Return to furnace after each reduction; soak at temperature for 10 minutes.

(6) After last pass, soak at temperature for 10 minutes and water quench.

Hot Rolling

Although hot rolling of VM-103 had been accomplished by NASA and Aeronutronic, no effort had been expended toward optimizing parameters of temperature, soaking time, maximum percent reductions, etc., nor was the importance of these parameters investigated. The goals were to establish parameters for maximum hot rolling reductions without significant edge cracking and without adversely affecting the microstructure (i.e., excessive grain growth, grain boundary carbide precipitation, etc.). A further goal was to investigate the variation in hot-workability between heats produced by VAR or ESR.

Samples of 1 x 1 in. (2.5 cm x 2.5 cm) square bar, representing both VAR and ESR as-forged material were subjected to rolling trials, using 5-50% reductions per pass at temperatures of 2100, 2175, and 2250°F (1150, 1190, and 1230°C) in order to ascertain maximum reductions without edge cracking. These results are discussed in Section 4.

Following this, in order to investigate effects of rolling temperature on microstructure and hardness, additional samples were hot rolled at each of these three temperatures to 0.10 in. (2.5 mm) thickness from the as-forged bar using an identical reduction schedule (Table II). After the last pass, the material was soaked at the rolling temperature for 10 minutes and water quenched. An average hardness was determined, and the resulting microstructure was observed by optical microscopy. The data, presented and discussed in Section 4, indicated that 2175°F (1190°C) was the optimum.

TABLE II

HOT ROLLING SCHEDULE USED TO ESTABLISH EFFECTS OF ROLLING TEMPERATURE ON MICROSTRUCTURE AND HARDNESS OF VM-103

Pass	Thickness Inch	Thickness mm	% Reduction	Reheat Time (Minutes)
1	0.90	22	10	10
2	0.79	20	12	8
3	0.68	17	14	8
4	0.56	14	17	8
5	0.45	11	20	8
6	0.31	8.0	30	8
7	0.22	5.6	30	5
8	0.15	3.9	30	5
9	0.10	2.5	35	

Notes:

1. Rolling Temperatures: 2100, 2175, 2250°F ± 25°F (1150-1190-1230°C ± 14°C)
2. Starting Material: 1 in. x 1 in. (25 x 25 mm) square bar
3. Initial Preheat Time: 30 minutes

rolling temperature. The 2175°F (1190°C) temperature and reduction schedule in Table II were utilized to produce additional 0.10 in. (2.5 mm) thick sheet for the remainder of the program.

Cold Rolling

Early NASA work and subsequent Aeronutronic efforts had indicated that VM-103 could be cold worked without much difficulty. Further efforts on this program were directed toward establishing base line parameters for producing sheet or foil by cold rolling, determining work hardening rates, and comparing effects of melting process (VAR vs. ESR) on cold workability. Small samples of hot rolled and annealed material from the four heats PF-11, PF-13, 20-1, and 20-5 were cold rolled ~5 - 40% to determine maximum reductions without significant edge cracking. The average hardness was measured after various reductions, and a hardness vs. percent cold work curve was established. Intermittent annealing schedules for cold rolled material were optimized as discussed below.

Annealing

In conjunction with the hot and cold rolling investigations, establishment of optimum annealing parameters (i.e., temperature, time, and cooling rate) was accomplished. The criteria for optimum annealing treatments were minimum hardness, minimum grain growth, and minimum matrix or grain boundary carbide precipitation. The following variables were investigated:

(1) Materials:

  (a) 0.10 in. (2.5 mm) thick hot rolled sheet from heats 20-5 and PF-11.

  (b) 0.10 in. (2.5 mm) thick cold rolled 25% reduced) sheet from heats 20-1, 20-5, PF-11, and PF-13.

(2) Temperatures: 2100, 2200, and 2300°F (1150, 1205, and 1260°C).

(3) Time: 30 minutes.

(4) Cooling Rates: water quench, air cool, and furnace cool.

Hardness and microstructure were observed on the heat treated samples. Using the criteria above, an annealing treatment of 2200°F (1205°C) for 30 minutes followed by a water quench was selected for both the hot and cold worked material. The data are presented in Section 4. These parameters were used throughout the program and unless otherwise specified were used for all "annealed" material.

Aging

Although VM-103 was designed to be a solid solution strengthened alloy, preliminary NASA data indicated an aging phenomenon, particularly in the 1600°F (870°C) range, resulting from precipitation of Co_3W associated with hcp cobalt stacking faults. Aging studies conducted on this program were directed toward achieving a better understanding of this effect and also determining if aging in conjunction with prior annealing or prior cold work would be useful as a strengthening mechanism.

Samples of annealed and 25% cold-worked 0.10 in. (2.5 mm) thick sheet from heat PF-11 were encapsulated in quartz tubes, evacuated to 10^{-5} mm Hg and sealed to prevent oxidation. The samples were then aged for periods of 1, 10, and 100 hours at temperatures of 700, 1000, 1300, and 1600°F (370, 540, 705, and 870°C). Hardness measurements, optical and electron microscopy, and extraction X-ray diffraction, were utilized to evaluate aging effects.

Mechanical Testing

Tensile Testing

The room temperature tensile properties of both annealed and cold worked material, and elevated temperature tensile properties of annealed material from all four heats were determined. Material which had been cold rolled from 0.10 to 0.040 in. (2.5 mm to 1.0 mm) sheet was utilized in the annealed and 15 and 25% cold worked conditions. Specimens were machined to the configuration shown in Figure 2 and Zyglo inspected. Testing was conducted on a 10,000 lb. capacity Instron testing machine equipped with a 2200°F (1205°C) resistance furnace. The specimens were brought from ambient to test temperature in about 30 minutes, soaked at temperature for an additional 15 minutes, and then tested at a strain rate of 0.005/minute to 0.4% offset yield followed by 0.05/minute to failure. An extensometer was used for measuring strain till about 1% elongation.

High Strain Rate Tensile Testing

Because of the desirability of utilizing VM-103 in the cold worked condition for short time elevated temperature applications and in order to determine effects of strain rate on mechanical properties, high strain rate tensile tests were performed.

Rectangular sheet specimens, 4 in. x 0.25 in. (100 mm x 6 mm) were fabricated from 0.040 in. (1.0 mm) thick sheet from heat PF-11 in the annealed 15% cold worked and 25% cold worked conditions. The specimens were tested on a "Gleeble" machine at a strain rate of 5/minute at temperatures of 75, 1800, 2000, and 2200°F (24, 980, 1095, and 1205°C). The samples were electrically self-resistance heated at a rate of approximately 500°F

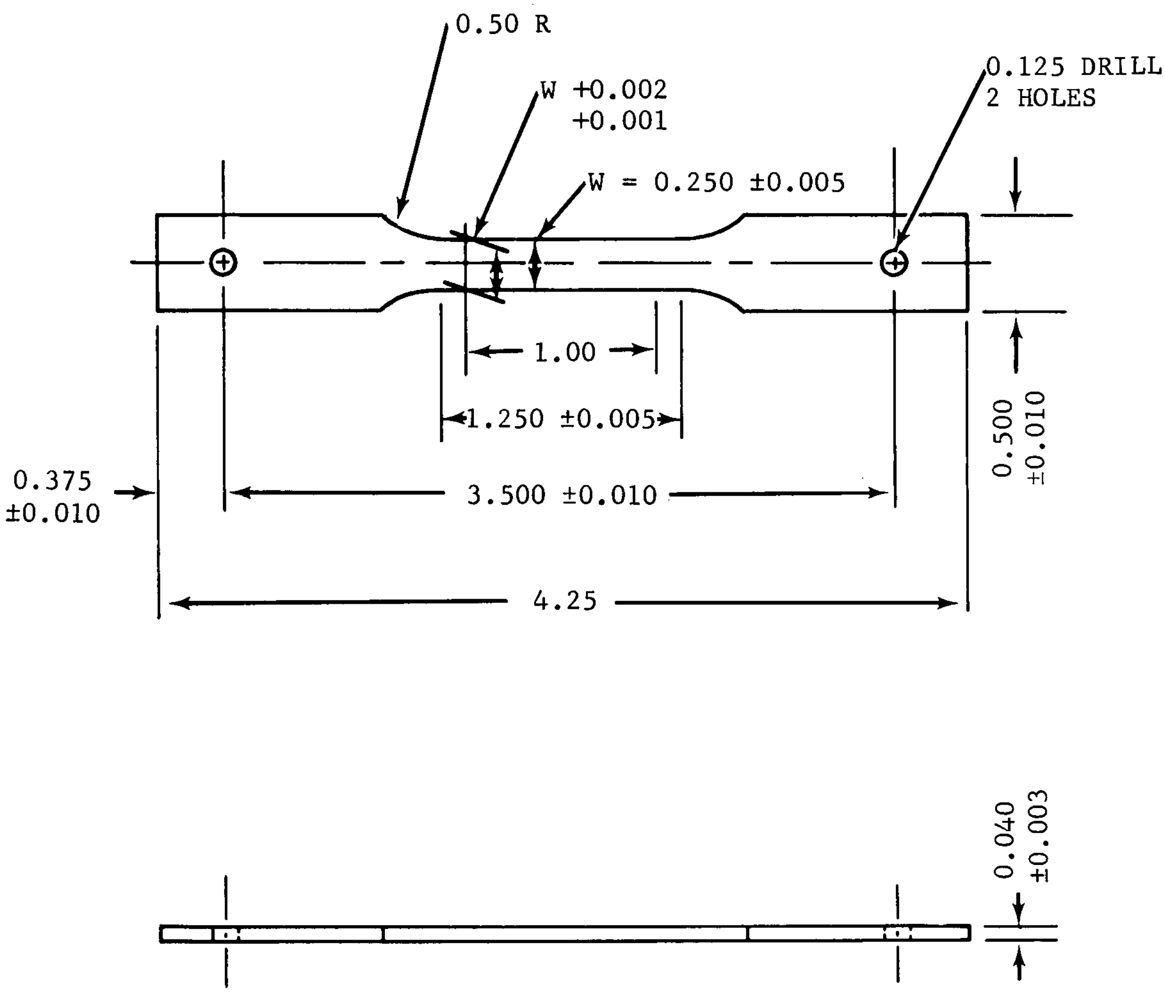


FIGURE 2. VM-103 SHEET TENSILE SPECIMEN
(All dimensions in inches)

(260°C)/sec., and held at temperature either 5 or 25 seconds prior to application of the load. Holding time was varied in an attempt to assess recovery and recrystallization behavior. The data were evaluated, and selected samples were examined metallographically.

Bend Testing

In order to ascertain the comparative cold forming characteristics of the two melting processes, bend tests were conducted on sheet from heats 20-1, 20-5, PF-11, and PF-13. Hot-rolled and annealed 0.10 in. (2.5 mm) sheet was cold reduced to 0.03 in. (0.76 mm) thickness and subsequently annealed using the parameters noted above. Bend specimens with a 20:1 width to thickness ratio were machined and tested in three point bending at 1T to 4T bend radii using ASTM E290-66 testing procedures.

Fatigue Testing

Tension-tension fatigue tests were conducted on VAR 20-1 and ESR PF-11 to compare the effect of melting process on fatigue properties. The test parameters of specimen thickness and stress were chosen to simulate missile hot gas valve thickness and cycle lives. Specimens were machined to the configuration shown in Figure 3 from material that had been cold rolled from 0.100 in. to 0.012 in. (2.54 mm to 0.30 mm) and then annealed. All testing was performed at 2000 cycles per minute on a Budd (Tatnall-Krause) VSP-150 fatigue testing machine with a direct stress attachment. The stress range was from 0 to 75 ksi (0 to 516 N/mm^2).

Metallurgical Analyses

Metallography

Samples were prepared for metallographic observation using the following method:

(1) Successive grinding on 180, 240, 360, and 600 grit silicon carbide discs.

(2) Polish on 6 micron followed by 1 micron diamond.

(3) Final polish on .05 micron alumina.

(4) Etch by swabbing for 4 to 8 seconds with hydrochloric acid saturated with ferric chloride.

(5) Ultrasonically clean for 2 to 3 minutes in distilled water.

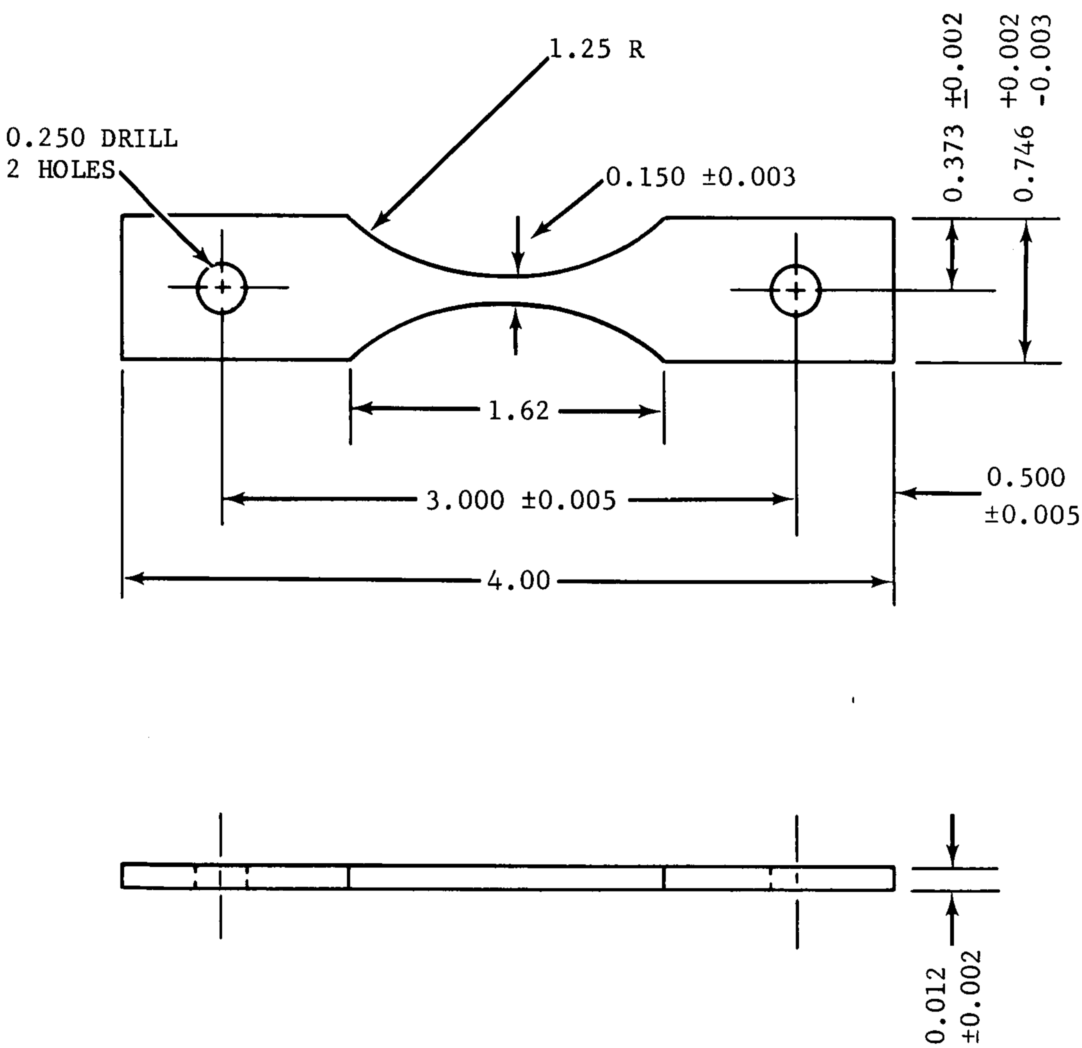


FIGURE 3. VM-103 FATIGUE SPECIMEN
(All dimensions in inches)

A Leitz MM-5 metallograph was used for optical microscopy and photomicrographs using bright field illumination. Observations were made at magnifications from 100 to 1000X. Grain size measurements were obtained using the ASTM E-112 linear intercept method. The reported grain sizes refer to the calculated "diameter" of an average grain with a mean standard deviation of ± 10%.

Electron Microscopy

Specimens were prepared for electron microscopy using disc samples and the jet technique. The equipment used including the photocell device to stop the polishing action upon specimen perforation has been described by DuBose and Stiegler.[7] Discs about 0.12 in. (3.0 mm) in diameter by 0.02 in. (0.5 mm) thick were dimpled using a room temperature 5% perchloric acid in glacial acetic acid electrolyte at 350 volts and about 200 ma/mm^2. The dimpled discs were then final electropolished at 0-5°C in a 10% sulfuric acid in ethanol electrolyte at 30-60 volts using rapid continuous stirring. Observations were made with a Hitachi HU-10 electron microscope operated at 100 kV.

Extraction X-Ray Diffraction Analysis

Phase Extraction Technique

A process for electrolytic dissolution of the cobalt alloy matrix leaving an undissolved precipitate residue for analysis was developed. The electrolyte consisted of 90% absolute methyl alcohol with 10% sulfuric acid. Samples, approximately 1 x 2 x 1/8 in. (25 x 51 x 3 mm), were weighed then clamped between two platinum cathodes slightly larger than the sample faces, placed 1/2 in. (13 mm) to each side of the sample faces. The sample was held in place with a strong alligator clamp. The clamp and platinum cathodes were firmly fastened to copper sheet strips which were rigidly held in place by mounting through a rubber stopper. The polishing was accomplished using a water jacket flask. The electrolyte was agitated gently by use of a magnetic stirrer. The bar was wrapped in Saran for easy removal of magnetic residues. Temperature was maintained throughout the process at 72°F (22°C). A constant voltage potential unit power source was used at 2.4 volts and 0.8 amperes. The residue was filtered and washed with clean electrolyte, then clean alcohol every two hours, at which time new electrolyte was put into the flask. Sufficient amounts of each sample were obtained in an 8 hour period, with three electrolyte changes, to allow for magnetic separations of the residues after they had been thoroughly rinsed and dried, then weighed along with the remainder of the unpolished sample.

X-Ray Diffraction Analysis

The separated portions were run on a Norelco X-ray diffractometer using filtered copper radiation set at 40 kilovolts and 20 milliamperes, with 1° scattering and receiving slits. Scans were made at 400 counts/second full scale, and intense lines were rerun at 800 or 1600 counts per second to prevent running off the chart scale.

Sample contents were approximated by using the sums of the (111) and (200) reflections of face centered cubic compounds and the (2000) and (0002) lines of the hexagonal compounds. All pairs of lines of each compound were added together, then each paired sum was divided by the total counts of all paired sums.

4. RESULTS AND DISCUSSION

Forging

The starting ESR ingots (Figure 1) were very sound and required only minor conditioning which was performed by hand grinding prior to forging. The VAR ingots exhibited moderate amounts of localized internal porosity. The approximate forging yields of all the ingots, calculated as the percent by weight of successfully forged material relative to the total weight of material submitted for forging, were: 20-1, 34%; 20-5, 100%; PF-11, 100%; PF-13, 90%; and PF-288, 98%. Total forging reductions were approximately 12:1 which equals or exceeds reductions usually performed in primary fabrication of production superalloy billets. A portion of the 20-5 1 in. x 1 in. (2.5 x 2.5 cm) bar was further forged to 1/2 in. x 1/2 in. (1.3 x 1.3 cm), further indicating good forgeability for as-cast material using hammer forging which is a relatively severe technique. Figure 4 shows 20-5 after 12:1 and 48:1 forging reductions. The forgeability compared favorably with other superalloys such as L-605.

The ESR ingots exhibited superior metal flow characteristics and less edge cracking than the VAR ingots. The high losses incurred in 20-1 were partially attributed to porosity within the ingot. After the last pass, the ingots were soaked at the forging temperature (2175°F or 1190°C) for 10 minutes and water quenched. In this condition, their hardness was R_c 34-37. The ESR and VAR billets exhibited mean grain sizes of about 25 and 35 microns, respectively. Photomicrographs taken transverse to the forging directions of the five heats are shown in Figure 5. Heat PF-288 (low Fe content) showed the best hot workability of all the ingots. Based on this work, the hot workability of VM-103, particularly ESR remelted material,

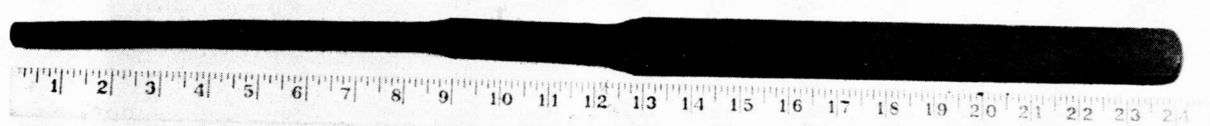
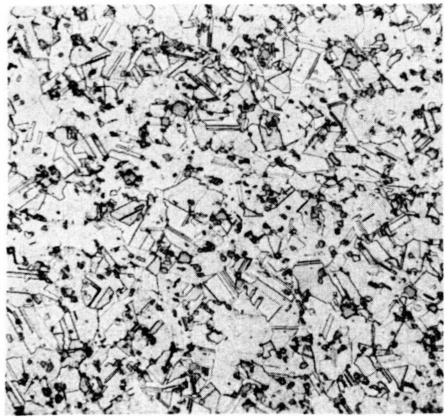
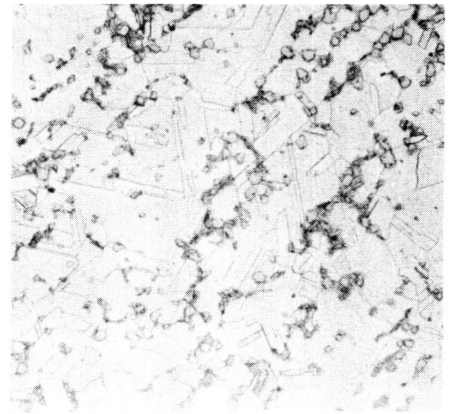


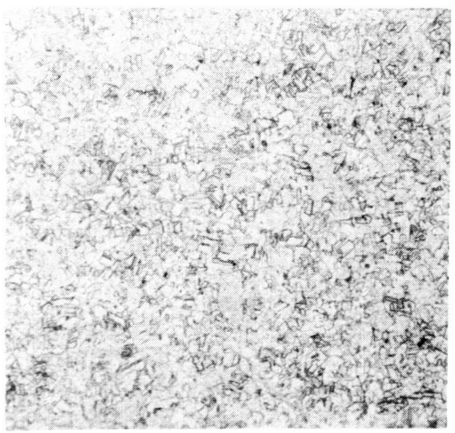
FIGURE 4. AS-FORGED VAR BILLET 20-5 ILLUSTRATING FORGING REDUCTIONS OF 12:1 and 48:1.



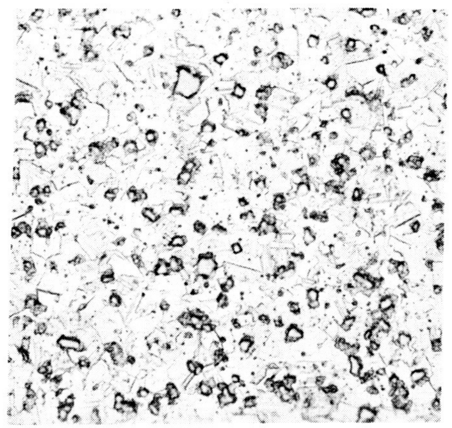
a. VAR 20-1 200X
R_c-35 G.S.-38 microns



b. VAR 20-5 200X
R_c-36 G.S.-37 microns



c. ESR PF-11 200X
R_c-34 G.S.-20 microns



d. ESR PF-13 200X
R_c-37 G.S.-23 microns

e. ESR PF-288 200X
R_c-39 G.S.-33 microns

FIGURE 5. TRANSVERSE SECTIONS OF AS-FORGED VM-103 HEATS.

appears comparable to or better than most other nickel and cobalt base superalloys. To summarize, typical parameters for forging VM-103 are:

Temperature: 2175°F (1190°C)

Reductions per pass:

- Hammer forging, cast structure ~10%
- Hammer forging, wrought structure ~20%

Hot Rolling

The criteria for selection of optimum rolling temperature and reduction schedule were trade-offs between (1) minimum edge cracking, (2) minimum grain growth, (3) minimum amounts of grain boundary and matrix precipitates, and (4) minimum as-rolled hardness. The maximum hot rolling reductions per pass without significant edge cracking at the three temperatures investigated are shown in Table III.

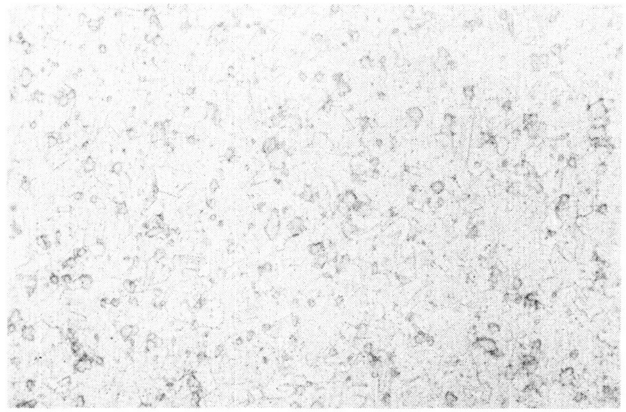
These data show that material from the ESR PF-11 heat could be reduced in significantly greater amounts per pass than material from the VAR 20-5 heat, again indicating better hot workability of ESR material. To optimize rolling temperature with respect to microstructure, samples were subsequently reduced identically from 1 in. (2.5 cm) thickness to 0.100 in. (0.25 cm) thickness at 2100, 2175, and 2250°F (1150, 1190, and 1230°C) as discussed in Section 3.

Microstructures of samples from ESR PF-11 and VAR 20-5 rolled with temperature being the only variable are given in Figures 6 and 7, respectively. As expected, metallographic observation indicated a slightly increasing grain size with increasing rolling temperature. The resulting grain sizes and R_c hardness values are shown in Table IV. (All reported R_c hardness values are the average from a minimum of six measurements producing a mean

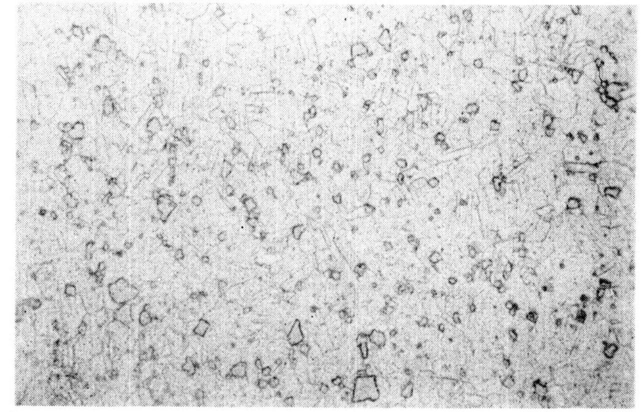
TABLE III

MAXIMUM HOT ROLLING REDUCTIONS PER PASS ACHIEVED WITHOUT EDGE CRACKING

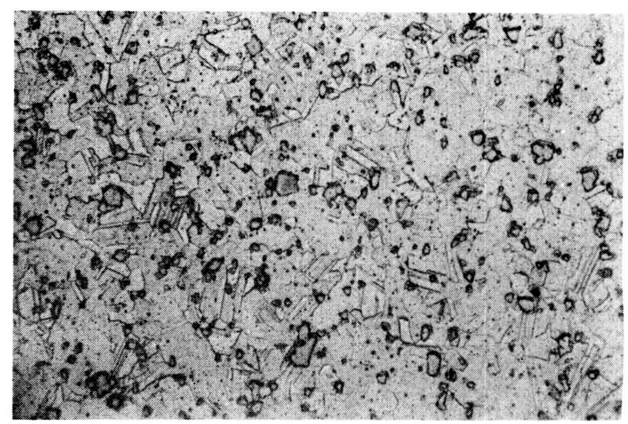
Temperature		Reduction (%)	
°F	°C	ESR PF-11	VAR 20-1
2100	1150	44	35
2175	1190	>44	>35
2250	1230	>50	50



a. 2100°F (1150°C) 500X
R_c-33 G.S.-10 microns

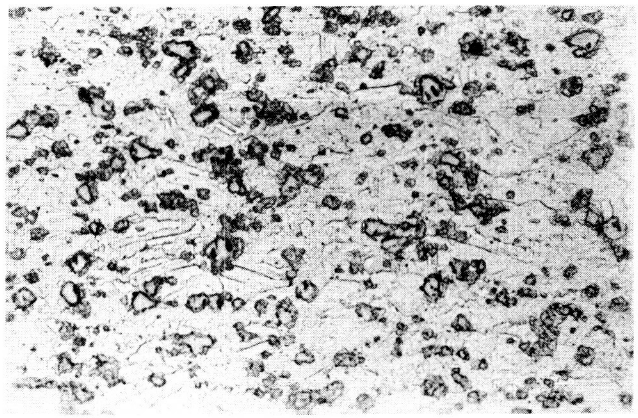


b. 2175°F (1190°C) 500X
R_c-33 G.S.-12 microns

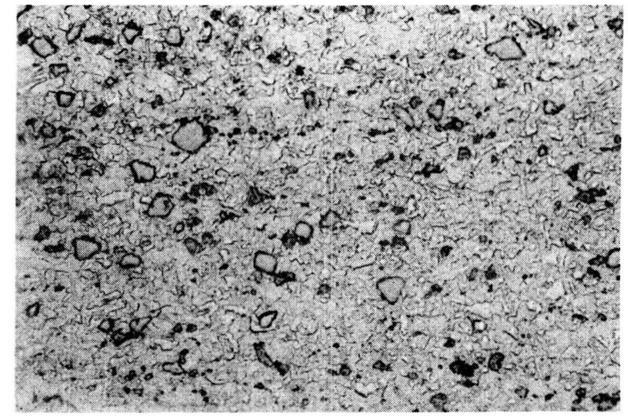


c. 2250°F (1230°C) 500X
R_c-31 G.S.-17 microns

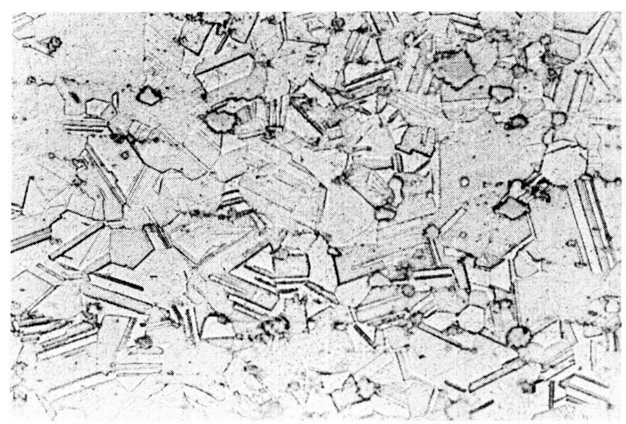
FIGURE 6. TRANSVERSE SECTION OF ESR PF-11. (Sheet Hot Rolled at the Indicated Temperatures.)



a. $2100^{\circ}F$ ($1150^{\circ}C$) 500X
G.S.-11 microns



b. $2175^{\circ}F$ ($1190^{\circ}C$) 500X
G.S.-9 microns



c. $2250^{\circ}F$ ($1230^{\circ}C$) 500X
R_c-36 G.S.-19 microns

FIGURE 7. TRANSVERSE SECTION OF HOT ROLLED VAR 20-5.
(Hot Rolled at the Indicated Temperatures.)

TABLE IV

EFFECT OF PROCESSING VARIABLES ON GRAIN SIZE AND HARDNESS OF VM-103

	VAR			ESR		
	Grain Size		Hardness	Grain Size		Hardness
Condition	(Microns)	(ASTM)	(R_c)	(Microns)	(ASTM)	(R_c)
As-Forged	38	6.5	36	20	8.2	34
Hot Rolled at 2100°F (1150°C)	11	10.0	--	10	10.2	33
Hot Rolled at 2175°F (1190°C)	9	10.6	--	12	9.7	33
Hot Rolled at 2250°F (1230°C)	19	8.5	36	17	8.7	31
Hot Rolled at 2175°F (1190°C) Annealed 30 Minutes at 2100°F (1150°C), Water Quenched	10	10.2	38	17	8.7	36
Hot Rolled at 2175°F (1190°C), Annealed 30 Minutes at 2200°F (1205°C), Water Quenched	8	10.9	38	14	9.3	34
Hot Rolled at 2175°F (1190°C), Annealed 30 Minutes at 2200°F (1205°C), Air Cooled	13	9.5	38	15	9.1	33
Hot Rolled at 2175°F (1190°C), Annealed 30 Minutes at 2200°F (1205°C), Furnace Cooled	13	9.5	46	10	10.2	36
Hot Rolled at 2175°F (1190°C), Annealed 30 Minutes at 2300°F (1260°C), Water Quenched	19	8.5	38	17	8.7	32

(Continued next page)

TABLE IV (Continued)

	VAR			ESR		
	Grain Size		Hardness	Grain Size		Hardness
Condition	(Microns)	(ASTM)	(R_c)	(Microns)	(ASTM)	(R_c)
Cold Rolled 25%, Annealed 30 Minutes at 2100°F (1150°C), Water Quenched	13	9.5	38	16	8.9	35
Cold Rolled 25%, Annealed 30 Minutes at 2200°F (1205°C), Water Quenched	14	9.3	36	14	9.3	34
Cold Rolled 25%, Annealed 30 Minutes at 2200°F (1205°C), Air Cooled	12	9.8	38	--	--	--
Cold Rolled 25%, Annealed 30 Minutes at 2200°F (1205°C), Furnace Cooled	12	9.8	46	--	--	--
Cold Rolled 25%, Annealed 30 Minutes at 2300°F (1260°C), Water Quenched	17	8.7	37	20	8.2	32

standard deviation of about ± 1 R_c.) The grain size of the alloy appears to be less sensitive to working temperature within this temperature range than that of other similar alloys such as L-605. As expected, the amount of precipitated carbides in the matrix was greater for material rolled at 2100°F (1150°C) than at 2175°F (1190°C) or 2250°F (1230°C). No grain boundary precipitation was noted at 1000X magnification for material rolled at any temperature. The hardness data in Table IV showed no significant effect of rolling temperature.

It was noted that ESR billet PF-11 had better workability characteristics than VAR billet 20-5. Although there was a large difference in as-forged grain size between these two billets (i.e., 20 versus 38 microns), this difference was minimized during hot rolling (12 versus 9 microns for rolling at 2175°F (1190°C)).

Completion of the hot rolling study resulted in the establishment of typical hot rolling parameters for VM-103 sheet, i.e.,:

Temperature: 2175°F (1190°C)

Reductions per pass:

- Forged billets 12-15%
- Previously hot rolled sheet 15-35%

Cold Rolling

As indicated in Section 3, cold rolling studies were performed to establish base line parameters for cold rolling sheet or foil, to establish work hardening rates, and to compare effects of melting process (VAR vs. ESR) on cold workability.

The maximum cold rolling reductions attainable without significant edge cracking for each of the four heats investigated along with resulting hardnesses were as shown in Table V. With the exception of heat PF-11 which showed little or no edge cracking at reductions less than 37%, the maximum nominal reductions per pass were 25% which produced a hardness of about Rockwell C-50. Heat PF-11, the most workable ESR heat available during that period of the program, showed no further hardening effects even at a 37% reduction. Heat PF-13 was the most difficult to cold roll and exhibited a tendency for very severe edge cracking at reductions greater than 25%. This may have resulted from a compositional effect, i.e., high Ti, a carbide former (as shown in Table I).

A percent cold work vs. hardness curve (Figure 8) was generated, which shows a rather rapid work hardening rate and a maximum hardness of approximately Rockwell C-52.

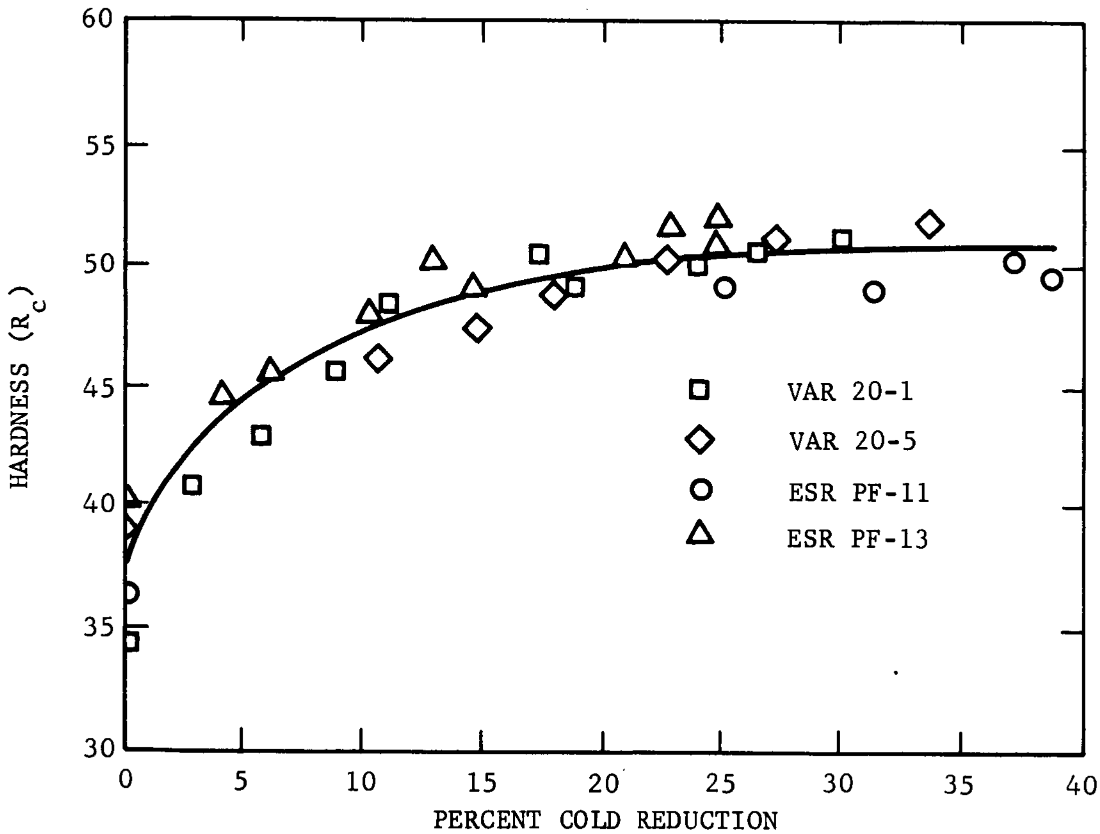


FIGURE 8. HARDNESS VS. PERCENT COLD REDUCTION OF VM-103.

TABLE V

MAXIMUM NOMINAL COLD ROLLING REDUCTIONS PER PASS WITHOUT EDGE CRACKING

Heat	Maximum Reduction (%)	As-Rolled Hardness (R_c)
VAR 20-1	25	50
VAR 20-5	25	50
PF-11	37	50
PF-13	25	51

Using nominal 25% maximum reductions and 2200°F (1205°C), 1/2 hour intermediate annealing treatments, samples of 0.012 in. (0.30 mm) thick foil were produced from all ESR and VAR sheet with a starting thickness of .01 in. (2.5 mm) with little or no difficulty. Again, ESR heat PF-11 appeared to be the most workable. The 0.012 in. (0.30 mm) thickness was selected as a severe test of cold workability and as a usable size since many missile hot gas valve components utilize superalloy foils in this thickness range.

Based on the above data and the annealing studies discussed below, a typical cold rolling schedule for VM-103 was established, i.e.,:

Nominal maximum reductions per pass: 25%

Intermediate annealing parameters: 2200°F (1205°C)
1/2 hour, water quench

Annealing

Selection of optimum annealing parameters for VM-103 was considered to be an important part of the development of the alloy, particularly in view of published data on L-605 (Co-15W-10Ni-20Cr superalloy). Data generated by Schulz on L-605 showed that 2150°F (1175°C) was superior to the conventional 2250°F (1230°C) with respect to grain size control and post aging ductility.[8] Harlow later confirmed the necessity of controlling grain size and grain boundary precipitates by adjusting annealing temperatures, times, and cooling rates for maximum cold workability.[9]

The criteria for selection of annealing parameters (temperature and cooling rate) for VM-103 were (1) minimum grain growth, (2) minimum amount of grain boundary and matrix precipitation, and (3) minimum hardness. The same criteria were utilized for both hot worked and cold worked material.

Because the effects of prior condition on grain growth, carbide precipitation, etc. were unknown, data were necessary to determine if different annealing parameters for each condition would be desirable.

As discussed in Section 3, hot-rolled and 25% cold-rolled samples of the VAR and ESR sheet material were subjected to annealing treatments of 1/2 hour at 2100°F, 2200°F, and 2300°F (1150°C, 1205°C, and 1260°C) and water quenched. Selected samples were heated similarly and air-cooled, or furnace-cooled to establish effects of cooling rate.

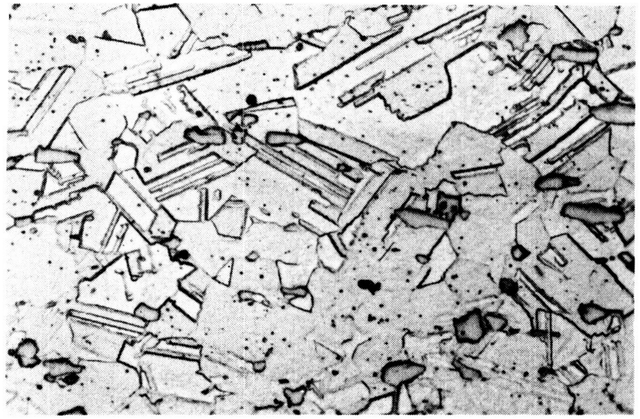
The grain size and hardness data resulting from these samples are presented in Table IV. The data indicate a very slight average decrease in hardness of 2-3 Rockwell C hardness numbers when the annealing temperature was raised from 2100°F (1150°C) to 2300°F (1260°C). This was true for both the hot and cold rolled ESR material, while the VAR material appeared to be less sensitive to annealing temperature. The annealed VAR material was several points harder in every case than the ESR.

Air cooling vs. water quenching resulted in virtually no effect on hardness, probably resulting from the relative rapid cooling rate achieved upon air cooling the thin sheet specimens. However, furnace cooling produced significantly higher hardnesses for prior hot-worked and prior cold-worked material as shown in Table IV. Optical microscopy showed no apparent explanation for this, since no differences were observed as shown in Figure 9. Further effort involving electron microscopy would be required to analyze this effect in greater detail.

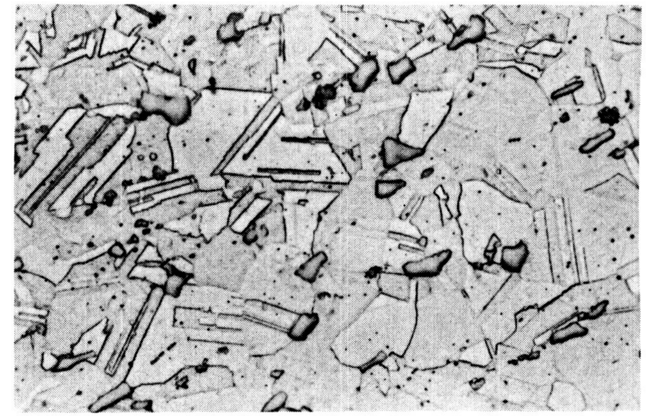
The effect of annealing temperature on grain size showed a slight grain growth with increasing temperature. This increase was approximately of the same magnitude for both the prior cold-worked and hot-worked sheet, which indicated that the same annealing parameters could be selected for each.

The microstructures of hot rolled and cold rolled sheet after annealing at 2100, 2200, and 2300°F (1150, 1205, and 1260°C) for 1/2 hour and water quenched are shown in Figures 10 and 11. The carbide distribution appeared insensitive to annealing temperature, with no evidence of undesirable precipitation in the grain boundaries. In general, the precipitates appeared somewhat smaller in prior cold-worked and annealed material than in the prior hot-worked and annealed material. This observation was made for both ESR and VAR material, indicating a possible finer dispersion and strengthening effect from intermediate cold or warm working. Further investigation of this phenomenon is recommended.

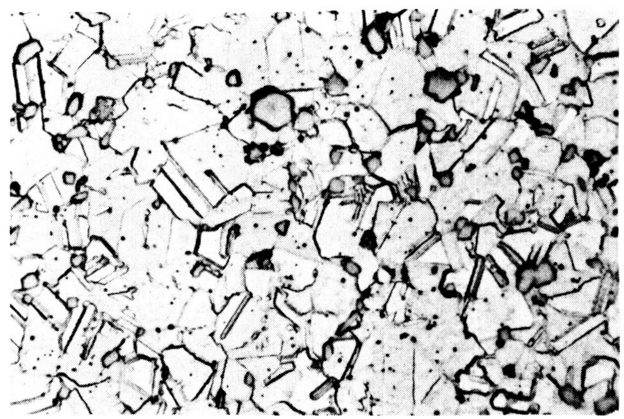
Based on the above hardness, grain size, and microstructure data and the fact that slightly more surface oxidation occurs at 2300°F (1260°C) than



a. Water Quenched — 1000X
R_c-34 — G.S.-14 microns



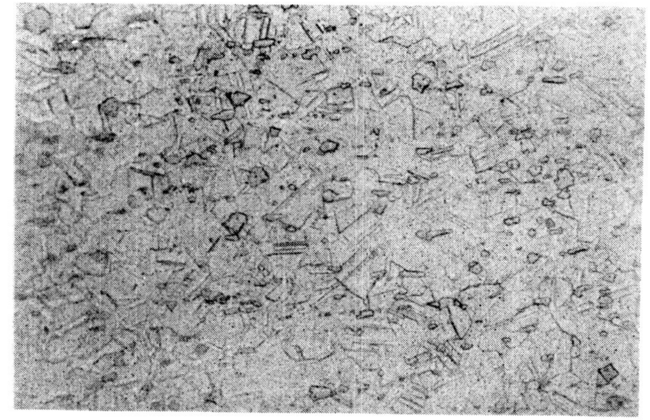
b. Air Cooled — 1000X
R_c-33 — G.S.-15 microns



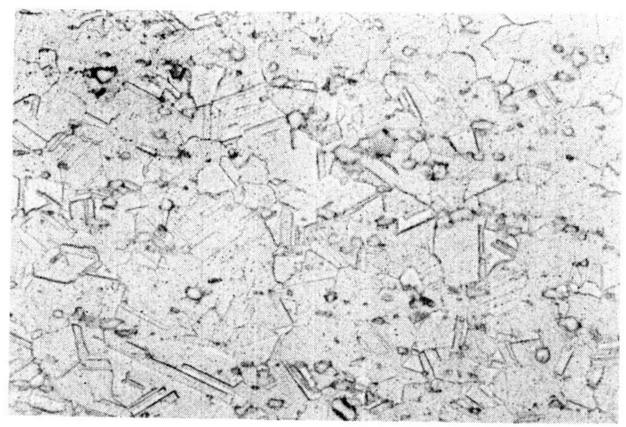
c. Furnace Cooled — 1000X
R_c-36 — G.S.-10 microns

FIGURE 9. MICROSTRUCTURE VS. COOLING RATE FROM 2200°F (1205°C). (ESR PF-11 Hot Rolled at 2175°F (1190°C), Annealed for 30 Minutes at 2200°F (1205°C) and Cooled as Indicated.)

a. 2100°F (1150°C) 500X
R_c-36 G.S.-17 microns

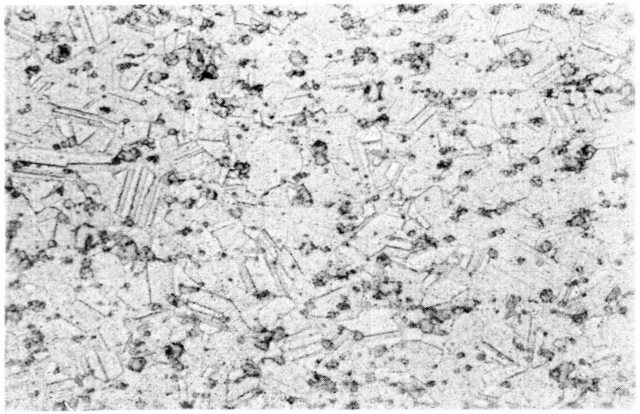


b. 2200°F (1205°C) 500X
R_c-34 G.S.-14 microns

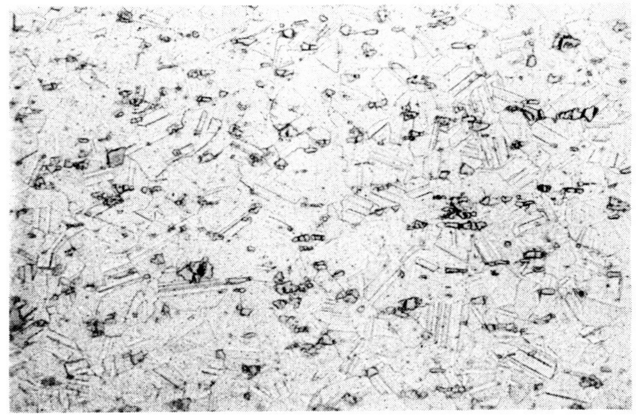


c. 2300°F (1260°C) 500X
R_c-32 G.S.-17 microns

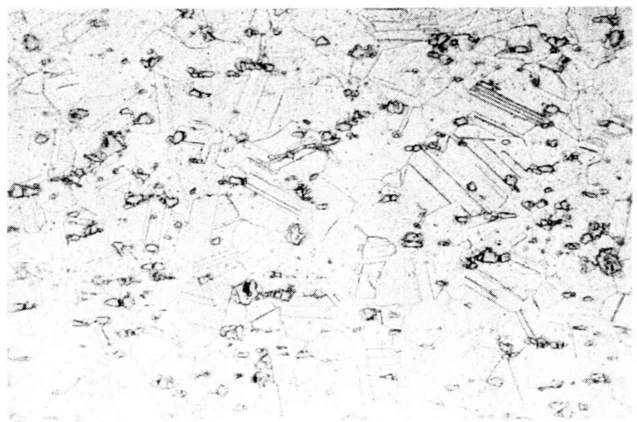
FIGURE 10. MICROSTRUCTURE VS. ANNEALING TEMPERATURE FOR HOT ROLLED VM-103. (ESR PF-11 Hot Rolled at 2175°F (1190°C), Annealed for 30 Minutes at the Indicated Temperatures and Water Quenched.



a. 2100°F (1150°C) 500X
R_c-35 G.S.-16 microns



b. 2200°F (1205°C) 500X
R_c-34 G.S. 14 microns



c. 2300°F (1260°C) 500X
R_c-32 G.S.-20 microns

FIGURE 11. MICROSTRUCTURE VS. ANNEALING TEMPERATURE FOR COLD ROLLED VM-103. (ESR PF-11 Cold Rolled 25%, Annealed for 30 Minutes at the Indicated Temperatures and Water Quenched.)

at the lower temperatures, an annealing temperature of 2200°F (1205°C) followed by a water quench was selected. Time at temperature was not investigated as a variable but would be expected to show little effect compared to differences in temperature. For sheet material, the 1/2 hour treatment utilized is probably more than adequate. Further work to optimize time as a variable annealing parameter may be desirable

Aging

As indicated previously, NASA had indicated an aging phenomenon in VM-103, particularly in the 1600°F (870°C) range, resulting from precipitation of a Co_3W phase associated with stacking faults in the hcp form of cobalt. The goal of the related effort on this program was to achieve a better understanding of this effect and to determine if aging would be useful as a strengthening mechanism.

Based on hot and cold workability, composition, and mechanical properties (reported below), ESR heat PF-11 was selected for the aging study. The 0.100 in. (2.5 mm) thick sheet samples representing annealed and 25% cold-worked sheet were encapsulated in quartz tubes and aged for 1, 10, and 100 hours at temperatures of 700, 1000, 1300, and 1600°F (370, 540, 705, and 870°C). Averaged hardness measurements after these various treatments are shown in Figure 12. Only one aging temperature, 1300°F (705°C), caused a response in the annealed sheet, causing an increase from Rockwell C-35 to C-43. A very small amount of precipitation could be seen at 1000X magnification on the prior-annealed sample aged at 1600°F (870°C) as shown in Figure 13. but not on those prior-annealed samples aged at the lower temperatures. In contrast, all the prior cold-worked samples responded to all the aging treatments in varying degrees, Figure 14. As can be seen, the slopes of the 700°F and 1000°F (370°C and 540°C) aging curves were still increasing after 100 hours, while overaging apparently occurred after about 10 hours at 1300 and 1600°F (705°C and 870°C). As shown in Figure 14, varying degrees of precipitation on the slip lines can be qualitatively correlated with hardness. These aging phenomena point to possible beneficial strengthening effects of thermomechanical processing.

Transmission electron microscopy was utilized on a very limited basis to examine these aging phenomena. As in optical metallography, fine precipitates were observed on slip lines in cold-worked and aged samples. Recovery of the 25% cold-worked material was incomplete after 100 hours at 1300°F (705°C) as indicated by transmission observations and diffuse broadened electron diffraction rings. Additional work in this area coupled with electron diffraction analysis for phase identification would be very worthwhile.

Extraction X-ray diffraction analysis to identify constituent phases of annealed, annealed and aged, and cold-worked and aged samples was also performed. The semiquantitative results are given in Table VI. Based on

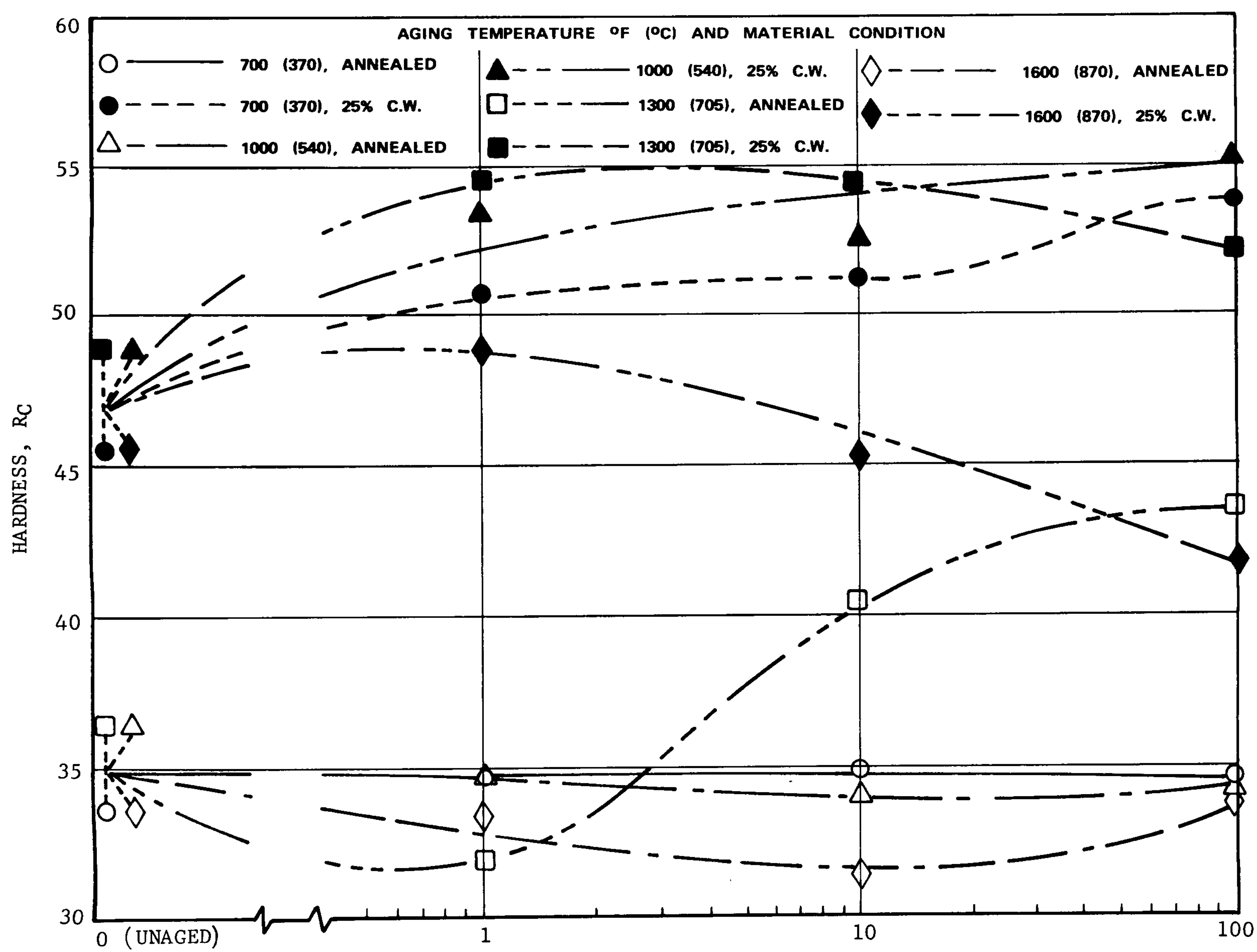
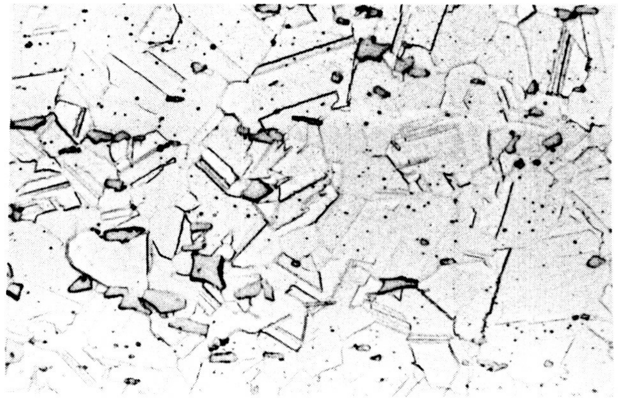
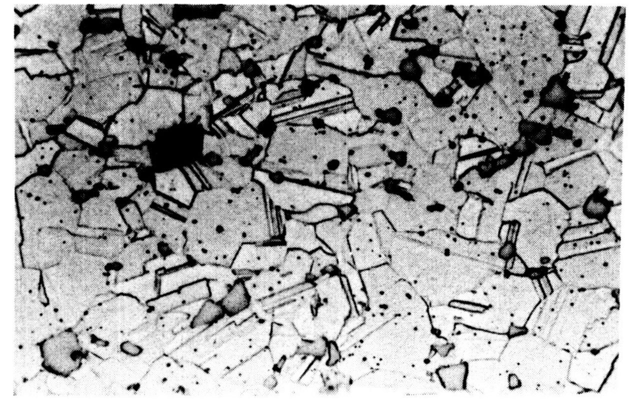


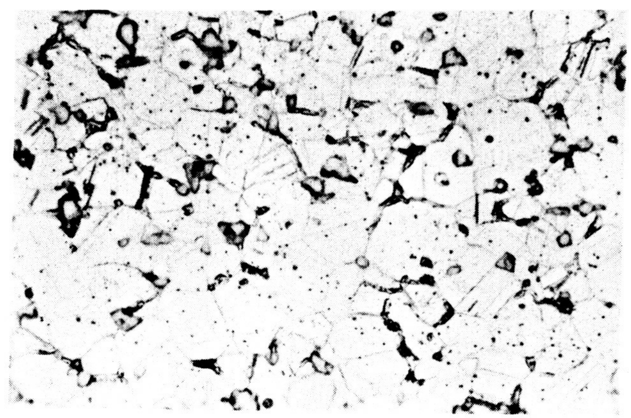
FIGURE 12. EFFECT OF AGING ON THE HARDNESS OF VM-103



a. 700°F (370°C) 1000X
G.S.-12 microns



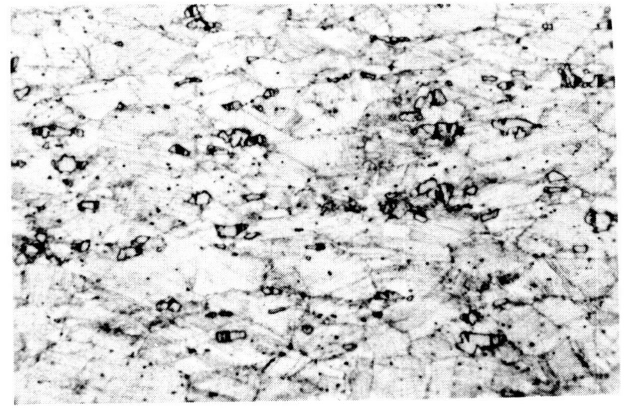
b. 1300°F (705°C) 1000X
G.S.-14 microns



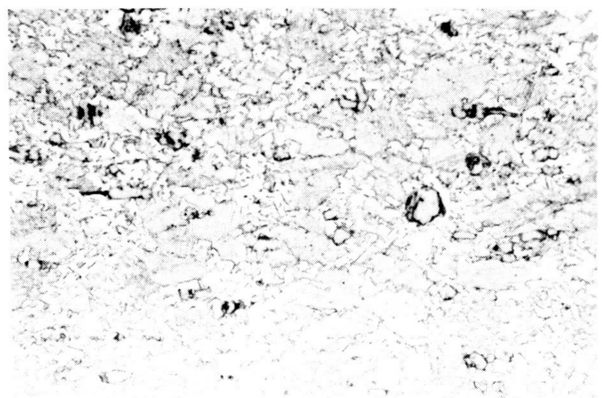
c. 1600°F (870°C) 1000X
G.S.-12 microns

FIGURE 13. EFFECT OF AGING ON MICROSTRUCTURE OF ANNEALED SHEET. (ESR PF-11, Annealed then Aged at the Indicated Temperatures for 10 Hours.)

a. 700°F (370°C) 1000X



b. 1300°F (705°C) 1000X



c. 1600°F (870°C) 1000X

FIGURE 14. EFFECT OF AGING ON MICROSTRUCTURE OF 25% COLD ROLLED SHEET. (ESR PF-11, 25% Cold Rolled, then Aged at Indicated Temperatures for 10 Hours.)

TABLE VI

EXTRACTION X-RAY DIFFRACTION ANALYSES OF VM-103

Condition	Phases Detected, Weight Percent					
	σ-M_6C	M_6C	Ti(C,N)	Zr(C,N)	f.c.c. Co_3W	hex Co_3W
Annealed	<0.1	<0.1	2.5	<0.1	2.3	---
Annealed + 1300°F(705°C) - 100 Hrs			3.2	0.1	3.6	---
25% C.W. + 700°F(370°C) - 100 Hrs			6.2	<0.1	0.7	---
25% C.W. + 1300°F(705°C) - 100 Hrs			8.7	<0.1	---	1.5
25% C.W. + 1600°F(870°C) - 100 Hrs			2.9	0.1	1.6	0.6

Lattice Parameters

Phase	Structure	a	c	c/a
σ-M_6C	Hexagonal	8.90	4.61	0.52
M_6C	Cubic	10.98	--	--
Ti(C,N)	f.c.c.	4.32	--	--
Zr(C,N)	f.c.c.	4.64	--	--
Co_3W	f.c.c.	3.57	--	--
Co_3W	Hexagonal	5.10	4.12	0.81

a correlation of these results with the hardness and tensile property results (presented below), it was concluded that the major aging strengthening mechanism is due to precipitation of the Co_3W phase which confirms NASA data. It is also possible that the overaging phenomenon includes a transformation from face centered cubic Co_3W to hexagonal Co_3W which has not been observed previously. A further investigation of this phenomenon is recommended.

Tensile Tests

Conventional tensile tests were conducted according to the procedure in Section 3 at 75, 1600, 1800, 2000, and 2200°F (24, 870, 980, 1095, and 1205°C) on hot rolled and annealed sheet from all five heats. In addition, room temperature tests were performed on 15 and 25% cold-worked sheet from VAR heats 20-1 and 20-5 and ESR heats PF-11 and PF-13. The goals were to establish room and elevated temperature properties of material produced by each melting process and to determine cold working effects on strength. In addition, as an exploratory effort to determine effects of aging on elevated temperature properties, 2200°F (1205°C) tests were performed on hot rolled sheet samples of ESR PF-11 after annealing at 1300°F (705°C) for 100 hours.

The data on annealed material are presented in Table VII and are plotted as the average of two samples in Figure 15. As can be seen, the annealed ESR material showed slightly higher yield and ultimate tensile strengths and generally higher elongations than the VAR material. The data generally confirm or are slightly better than preliminary NASA data on small induction melted laboratory heats.

The data in Table VII show that the room temperature yield strength was significantly increased by 15% cold work and nominally doubled by 25% cold work. The ultimate strengths showed a smaller percentage increase, and the elongations were significantly reduced. The ESR heat, PF-11, showed the highest ductility of all heats both in the annealed and cold-worked conditions. The results indicate the desirability of considering cold-worked VM-103 for use in applications requiring high strengths at low or intermediate temperatures, or even at high temperatures for short periods of time.

As shown in Table VII, the 1300°F (705°C) 100 hour aging treatment was effective in raising the 2200°F (1205°C) yield strength from 4.2 to 10.1 ksi (29 to 70 N/mm^2) and in raising the ultimate strength from 7.6 to 10.4 ksi (52 to 72 N/mm^2) while lowering the ductility from 97 to 68%. This 140% increase in yield strength indicates that additional work should be performed in efforts to further improve elevated temperature strength for relatively short time applications, perhaps by thermomechanical processing.

TABLE VII

TENSILE PROPERTIES OF VM-103 HEATS

Heat	Condition	Test Temperature °F	Test Temperature °C	0.2 Yield Strength ksi	0.2 Yield Strength N/mm^2	Ultimate Tensile Strength ksi	Ultimate Tensile Strength N/mm^2	Elongation in 1 In. (2.5 cm) %
20-1 (VAR)	Annealed	75	24	93	640	149	1030	16
20-5 (VAR)	Annealed	75	24	90	620	142	979	17
PF-11 (ESR)	Annealed	75	24	84	580	159	1100	40
PF-13 (ESR)	Annealed	75	24	103	710	167	1150	12
PF-288 (ESR)	Annealed	75	24	97	670	164	1130	21
20-1 (VAR)	15% C.W.	75	24	145	1000	202	1390	5
20-5 (VAR)	15% C.W.	75	24	140	965	195	1340	4
PF-11 (ESR)	15% C.W.	75	24	158	1090	191	1320	12
PF-13 (ESR)	15% C.W.	75	24	205	1410	242	1670	2
20-1 (VAR)	25% C.W.	75	24	188	1300	250	1720	3
20-5 (VAR)	25% C.W.	75	24	200	1380	260	1790	3
PF-11 (ESR)	25% C.W.	75	24	178	1230	246	1700	6
PF-13 (ESR)	25% C.W.	75	24	194	1340	248	1710	3
20-1 (VAR)	Annealed	1600	871	56	390	80	550	2
20-5 (VAR)	Annealed	1600	871	48	330	72	500	4
PF-11 (ESR)	Annealed	1600	871	46	320	57	390	1
PF-13 (ESR)	Annealed	1600	871	51	350	85	590	3
PF-288 (ESR)	Annealed	1600	871	65	450	85	590	4
20-1 (VAR)	Annealed	1800	982	26	180	46	320	3
20-5 (VAR)	Annealed	1800	982	22	150	44	300	5
PF-11 (ESR)	Annealed	1800	982	22	150	35	240	9
PF-13 (ESR)	Annealed	1800	982	24	170	43	300	10
PF-288 (ESR)	Annealed	1800	982	28	190	39	270	31

(Continued on next page)

TABLE VII (Continued)

Heat	Condition	Test Temperature		0.2 Yield Strength		Ultimate Tensile Strength		Elongation in 1 In. (2.5 cm)
		°F	°C	ksi	N/mm^2	ksi	N/mm^2	%
20-1 (VAR)	Annealed	2000	1093	9.0	62	>18	>120	--
20-5 (VAR)	Annealed	2000	1093	6.5	45	15	100	16
PF-11 (ESR)	Annealed	2000	1093	11	76	20	140	15
PF-13 (ESR)	Annealed	2000	1093	9.0	62	19	130	21
PF-288 (ESR)	Annealed	2000	1093	13	90	33	230	39
PF-11 (ESR)	Annealed	2200	1204	4.2	29	7.6	52	97
PF-288 (ESR)	Annealed	2200	1204	4.1	28	7.8	54	71
PF-11 (ESR)	Annealed + aged at 1300°F (705°C) for 100 hours	2200	1204	10.1	70	10.4	72	68

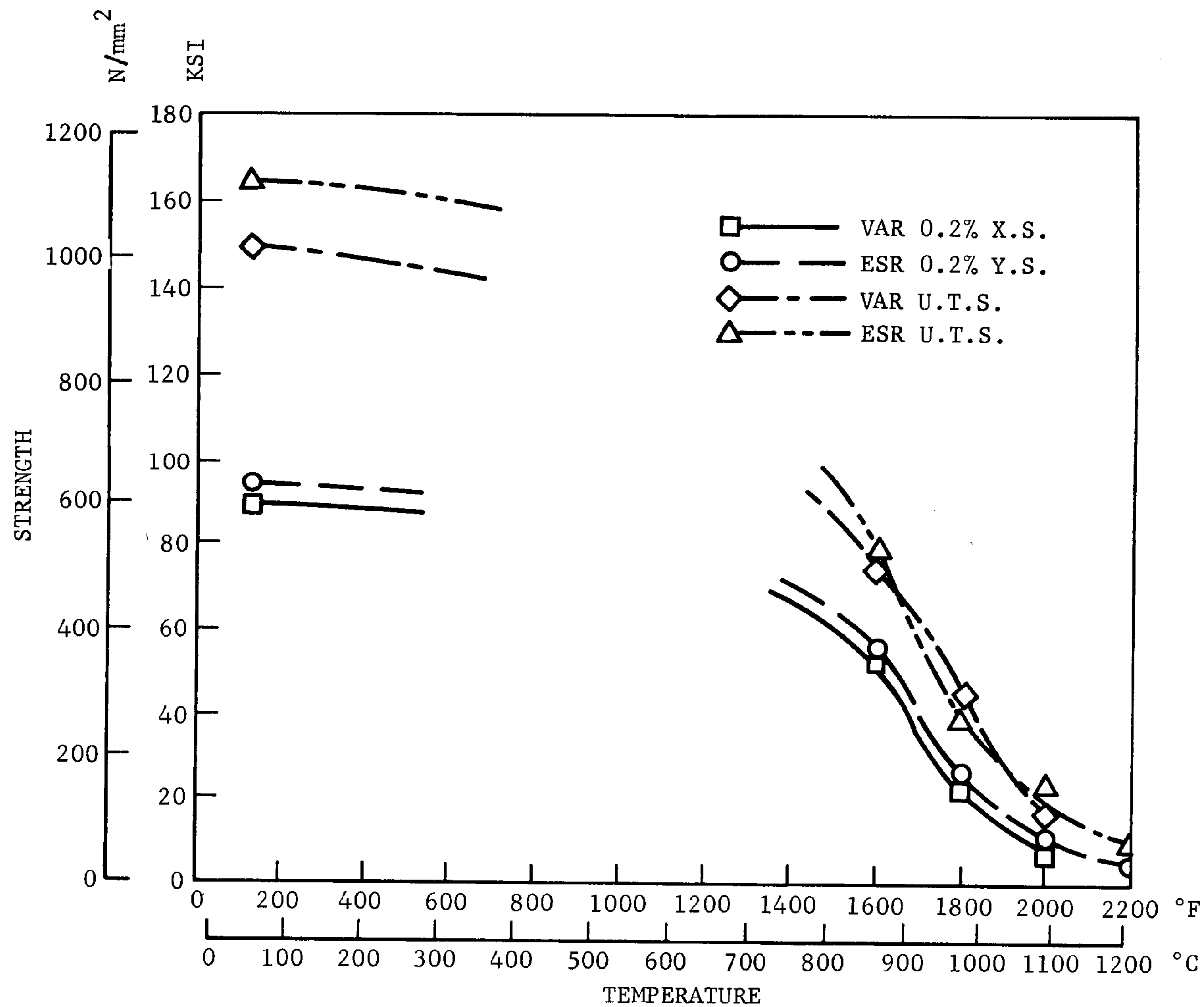


FIGURE 15. TENSILE STRENGTH VERSUS TEST TEMPERATURE FOR ANNEALED VM-103 SHEET.

High Strain Rate Tensile Tests

To investigate the effect of strain rate and to determine very short time elevated temperature tensile properties, high strain rate tests were conducted according to the procedure noted in Section 3. Samples of annealed, 15% cold-worked and 25% cold-worked sheet from ESR heat PF-11 were tested at 75, 1800, 2000, and 2200°F (24, 980, 1095, and 1200°C) at a strain rate of 5/minute.

The data, presented in Table VIII, show that VM-103 is very strain rate sensitive. The yield strength data for all temperatures were at least two times higher than the conventional strain rate results (Table VII).

The elevated temperature data show that at 1800°F (980°C) the recovery process was more sluggish for 15% than for 25% cold-worked material. This difference in kinetics as a function of percent prior cold deformation became insignificant at 2200°F (1205°C). However, at 2000°F (1095°C) the annealed material still showed higher strengths than the cold-worked material. With the exception of the 15% cold-worked specimen tested at 2200°F (1205°C), the data indicated an increase in strength with soaking time at temperature, indicating a possible rapid aging process.

Selected samples were examined by optical metallography after testing. These indicated, as expected, more complete recrystallization with increasing testing temperature and soaking time as shown in Figure 16.

Bend Tests

In order to assess the cold forming characteristics of ESR vs. VAR material, bend tests were conducted on 0.030 in. (0.76 mm) sheet from ESR heats PF-11 and PF-13 and VAR heats 20-1 and 20-5 according to the procedure in Section 3. The results in Table IX indicated that VAR heat 20-1 exhibited the poorest bend ductility; VAR 20-5 and ESR PF-13 were comparable, and ESR PF-11 was far superior. These data supported the previously observed superior hot and cold workability and ductility of ESR PF-11.

Fatigue Testing

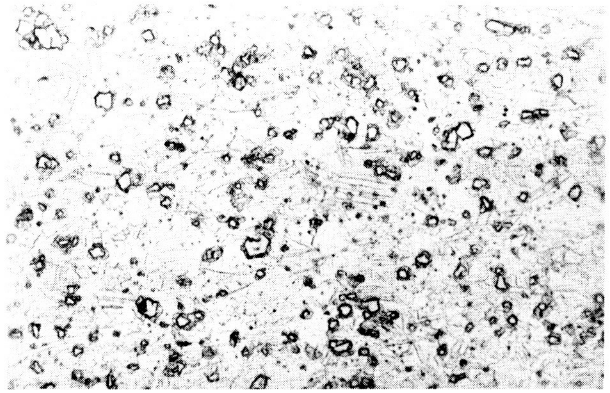
Tension-tension fatigue specimens were tested as discussed in Section 3 primarily for purposes of determining differences in fatigue behavior of ESR vs. VAR material. No attempt was made to generate an S/N curve. The data shown in Table X were very scattered but when averaged indicated a slight superiority of the ESR heat. More work would be required using standard F and t statistical tests in order to generate more reliable conclusions.

TABLE VIII

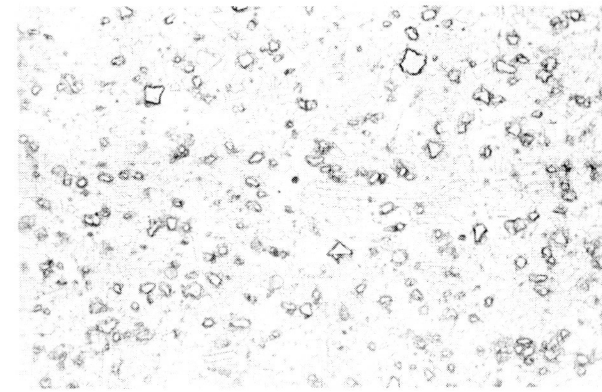
HIGH STRAIN RATE* TENSILE PROPERTIES OF ESR HEAT PF-11

Condition	Test Temperature (±5°F) °F	°C	Time at Temperature, Sec.	0.2 Yield Strength ksi	N/mm^2	Ultimate Tensile Strength ksi	N/mm^2	Elongation in 0.8 In. (2.0 cm) %	Reduction of Area %
15% C.W.	75	24	--	190.0	1,310	211.7	1,460	0.3	--
25% C.W.	75	24	--	202.3	1,946	208.4	1,437	0.3	--
25% C.W.	75	24	--	194.4	1,340	213.0	1,469	0.3	--
Annealed	75	24	--	158.0	1,089	175.0	1,207	0.4	--
15% C.W.	1800	982	5.0	--	--	50.6	349	15.0	63
15% C.W.	1800	982	25.5	48.5	334	55.7	384	16.3	62
25% C.W.	1800	982	4.8	38.5	265	44.1	304	18.8	53
25% C.W.	1800	982	15.1	42.9	296	51.8	357	17.5	55
Annealed	1800	982	5.0	40.3	279	49.2	339	20.0	47
Annealed	1800	982	25.6	40.2	277	50.1	345	22.5	51
15% C.W.	2000	1093	5.1	24.9	172	28.1	194	25.0	53
15% C.W.	2000	1093	25.2	27.3	188	29.0	200	23.8	52
25% C.W.	2000	1093	5.1	25.0	172	27.9	192	27.5	50
25% C.W.	2000	1093	25.3	27.4	189	28.3	195	26.0	60
Annealed	2000	1093	25.2	30.5	210	31.5	217	28.5	72
15% C.W.	2200	1204	5.0	18.5	128	18.7	129	23.7	54
15% C.W.	2200	1204	25.8	17.8	123	17.9	123	22.5	51
Annealed	2200	1204	5.0	18.3	126	18.5	128	22.5	53
Annealed	2200	1204	25.7	18.4	127	18.8	130	30.0	82

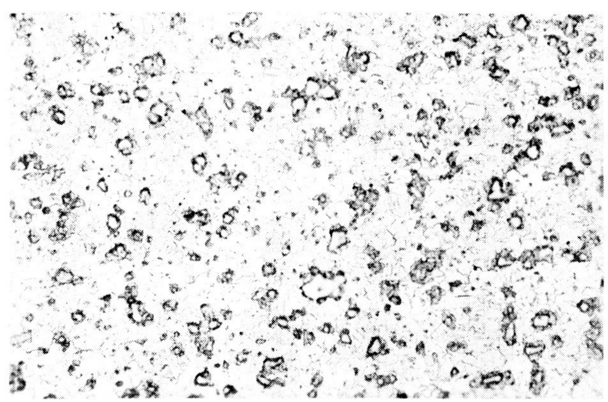
*Strain Rate: 5/min.



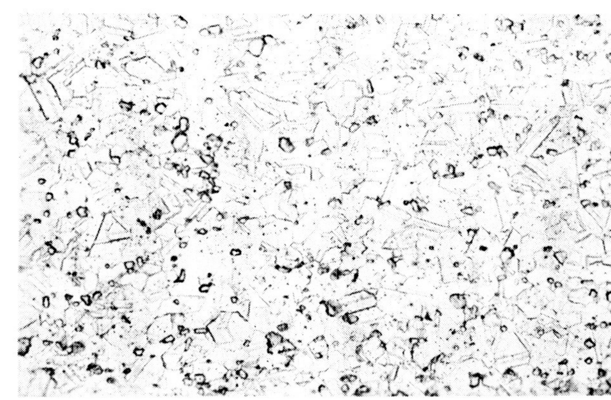
a. 1800°F (980°C) 500X



b. 1800°F (980°C) 500X



c. 2000°F (1093°C) 500X



d. 2200°F (1204°C) 500X

FIGURE 16. EFFECT OF TESTING TIME AND TEMPERATURE ON MICROSTRUCTURE OF 15% COLD WORKED ESR PF-11 SHORT TIME ELEVATED TEMPERATURE, HIGH STRAIN RATE TENSILE SPECIMENS. (The Microstructures are Representative of the Uniform Elongation Section of the Gage Length. Figure a was at Temperature for aTotal of 5 Seconds while Figures b, c, and d were Exposed for 25 Seconds.)

TABLE IX

MINIMUM BEND RADII OF VM-103 HEATS

Heat	Minimum 90° Bend Test Radius Without Cracking
VAR 20-1	>4T
VAR 20-5	4T
ESR PF-11	1T
ESR PF-13	4T

Note: T refers to thickness of the specimen which was 0.030 in. (0.76 mm).

TABLE X

TENSION-TENSION FATIGUE TEST RESULTS

	Total Cycles to Failure	
	VAR 20-1	ESR PF-11
	37,000	212,400
	576,000	18,600
	82,100	15,100
	130,900	138,000
	100,100	676,800
	25,300	---
Averages:	158,600	212,200

Note: Samples were stressed 0 to 75 ksi (0 to 520 N/mm^2)

5. SUMMARY OF RESULTS AND RECOMMENDATIONS

Based on the results of this VM-103 superalloy development and metallurgy study, the following conclusions and recommendations were made:

(1) VM-103 can be melted and fabricated by production oriented processes including vacuum arc or electroslag remelting, hammer forging, hot-rolling and cold-rolling, and cold forming. The properties of material produced from 25-50 lb. (11-23 kg) heats using these processes with optimum parameters developed on this program are comparable or somewhat better than achieved on 3-4 lb. (<2 kg) laboratory heats.

(2) VM-103 appears to be competitive with conventional nickel and cobalt base superalloys in fabricability and in elevated temperature properties at or above 1800°F (980°C). It is particularly attractive as a candidate for short time high temperature applications.

(3) Electroslag remelted VM-103 reveals better hot and cold workability, higher tensile properties, and higher ductility than vacuum arc remelted material.

(4) VM-103 work hardens rapidly with a corresponding increase in strength and hardness. The increase in strength is retained for short times at temperatures as high as 1800°F (980°C).

(5) The alloy is somewhat age hardenable due to precipitation of a Co_3W phase in the annealed condition and to a greater degree after cold working. Preliminary data indicated a 140% increase in 2200°F (1205°C) yield strength as a result of aging prior annealed material. Thermomechanical processing investigations are recommended as a means for enhancing alloy properties.

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