ILIKON CORPORATION
NATICK, MASSACHUSETTS

BASIC STUDIES ON

DISPERSION HARDENING

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QUARTERLY REPORT

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1. Introduction

During the quarter currently being reported (12 July - 11 October, 1964) work has continued along the lines indicated in the previous quarterly report. Research has been carried out in three fields. These are:

(1) The measurement of strains using transmission electron microscopy;

(2) The measurement of strains using x-ray diffraction;

(3) The effect of high temperature anneals on internal structure and, in particular, on agglomeration.

2. Transmission Electron Microscopy

In our previous attempts at observing the diffraction effects caused by the presence of elastic strains at the particle-matrix interface, we were only able to see them in association with very small particles. As will be discussed later, it is reasonable to assume that these strains are due to the existence of coherency across the particle-matrix interface. Elastic strains across this interface could also be caused by the difference in the coefficient of thermal expansion of the particle and matrix. All the specimens which we examined prior to the period being reported were slowly cooled from temperatures in the range 600-800^{o}C before being

examined by transmission electron microscopy. It is probable that the strains introduced into these specimens, by the difference in coefficient of thermal expansion, were to a large extent relieved by diffusional processes during this slow cooling. It was therefore decided to build an inert atmosphere, vertical tube furnace in which we could rapidly quench specimens from temperatures in the range 600°C - 800°C.

The specimens used were of TD nickel (Ni + 2% ThO_2), which was obtained from Dupont in the form of ½" diameter extruded rod. Slices .050" thick were cut from this rod and then rolled down to .015". After being given a softening anneal of 1 hour at 650°C, these pieces were once again rolled and .005" thick foil was thus obtained. This foil was suspended in the hot zone of the vertical furnace (Figure 1) and the temperature raised to either 600°C, 700°C or 800°C. The foil was maintained at this temperature for ½-hour and then dropped rapidly, by using a heavy electrical current to burn out the suspending wire, into a glycerine bath at the ambient temperature. The foils thus obtained were thinned down in the normal manner and examined by transmission electron microscopy. No difference could be detected between the quenched foils and the slowly cooled foils. If quenching does introduce strain, it is within the undetectable range. We do not believe that this is due to our techniques of observation as we know we can see strains due to coherency.

The magnitude of the diffraction effect (D-shaped lobe) for which we are looking is proportional to the magnitude of the elastic

strain. If the elastic strain is small, it is possible that the diffraction effect is too small to be detected. We are, therefore, about to start exploring ways of introducing larger elastic strains due to the difference in coefficient of thermal expansion. The magnitude of this strain is dependent on three factors. These are:

(1) The difference in coefficient of thermal expansion of the particle and of the matrix.

(2) The range of temperature through which the specimen is cooled.

(3) The extent to which the strains are relieved, by diffusional and other processes, during the cooling. It would be expected that a more rapid quench would give greater retention of the elastic strain.

In TD nickel, the coefficients of expansion are approximately:

ThO_2	$8.5 \times 10^{-6}/^{\circ}C$
Ni	$12.8 \times 10^{-6}/^{\circ}C$

The difference is, therefore, approximately $4.3 \times 10^{-6}/^{\circ}C$. If we consider the Ni-$SiO_2$ system (where the SiO_2 is in the amorphous form (tridymite) - coefficient of expansion $0.5 \times 10^{-6}/^{\circ}C$), then the difference in coefficient of expansion is approximately $12.3 \times 10^{-6}/^{\circ}C$ - almost three times as large. We would, therefore, expect to be able to see effects due to quench-induced strain more easily in the Ni-SiO_2 system. A further supply of

this alloy will, therefore, be obtained. Of the other two factors, the rate of quenching is the most important. If we raise the temperature from which we quench we gain nothing unless we quench sufficiently rapidly to prevent relaxation, due to diffusion, of the strains. Consideration is therefore being given to ways of more rapidly quenching the specimen.

The other source of interfacial strain with which we are concerned is that due to the existence of coherency across the particle-matrix interface. In a previous report we described how we think a particle is formed and how this coherent interface comes about. Associated with this interface there is strain energy and, as the particle increases in size, the amount of strain energy due to the existence of coherency increases until, above a certain particle, it is energetically more favorable to have an incoherent interface. We can, therefore, expect to see coherency strains associated with only the smaller particles. This is, in fact, what we have observed. To be able to make a study of these strains it is necessary to observe material which contains large numbers of small particles (100Å or less). Such material is at present being obtained.

3. The Measurement of Strain Using X-Ray Diffraction

The work on the measurement and analysis of the profile of x-ray diffraction lines, which we started in the last quarter, has been continued. It soon became evident that the standard specimen holder which we were using did not allow us to position

a specimen with sufficient reproducibility to make the extremely precise measurements which this work requires. A new specimen holder, designed specifically for use with specimens in the form of flat plates, was therefore designed and built. This has proved to be adequate. A further delay then occurred when it was found that some part of the counting equipment attached to our Picker x-ray diffraction unit had turned unstable. Picker are at present repairing this equipment and we anticipate that we will start obtaining results in the very near future.

As has been previously reported, we have written and checked out the computer program which we will use to analyze these results when we do obtain them and we have prepared the specimens on which we will carry out these measurements.

Two different series of experiments will be carried out into the causes of line broadening. In the first, a series of Ni + 1% Cr specimens, in the form of well annealed flat plates, will be internally oxidized under various conditions, and the shape of the x-ray diffraction lines analyzed in order to see if we can detect any elastic strains which have been introduced by this process. These results will then be compared with the structure of the alloy as determined by transmission electron microscopy. Figure 2 shows the structure of a thin foil of Ni + 1% Cr which was internally oxidized at the same time as one of the flat plates on which we propose to carry out the line broadening experiments.

The second series of experiments will be carried out into the changes in internal structure which occur during the anneal-

ing of cold worked TD nickel. It has been reported[1] that TD nickel undergoes almost complete recovery on annealing at temperatures above 600°C. During our attempts at observing the D-shaped lobes associated with interfacial strains, we observed that this was not necessarily so and that even after a 1 hour anneal at 700°C considerable dislocation structure could be seen in certain areas. Plates of TD nickel which had been annealed for 1 hour at temperatures above 600°C were used in line broadening experiments. A steady decrease in the width of the diffraction line with increase in annealing temperature was observed. The observations which we have carried out so far have been of a preliminary nature and further systematic work in this field is required before we can draw any definite conclusions.

4. Diffusion and Agglomeration

The preparation of specimens of TD nickel and of pure Ni, on which we propose to carry out diffusion studies, has been carried out. These specimens will now be plated with radioactive Ni 63 and the diffusion rates at various temperatures will be measured. The purpose of these experiments is to see if the presence of the particles and the associated dislocation structure has any effect on the diffusion kinetics. If they do, then we will see if this enhanced diffusion can explain the rates of agglomeration which we observe at high temperatures.

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(1) "Recrystallization Behavior of Cold Rolled TD-Nickel", M. C. Inman, K. M. Zwilsky and D. H. Boone, ASM Trans. Quarterly, Vol. 57, p. 701 (1964).

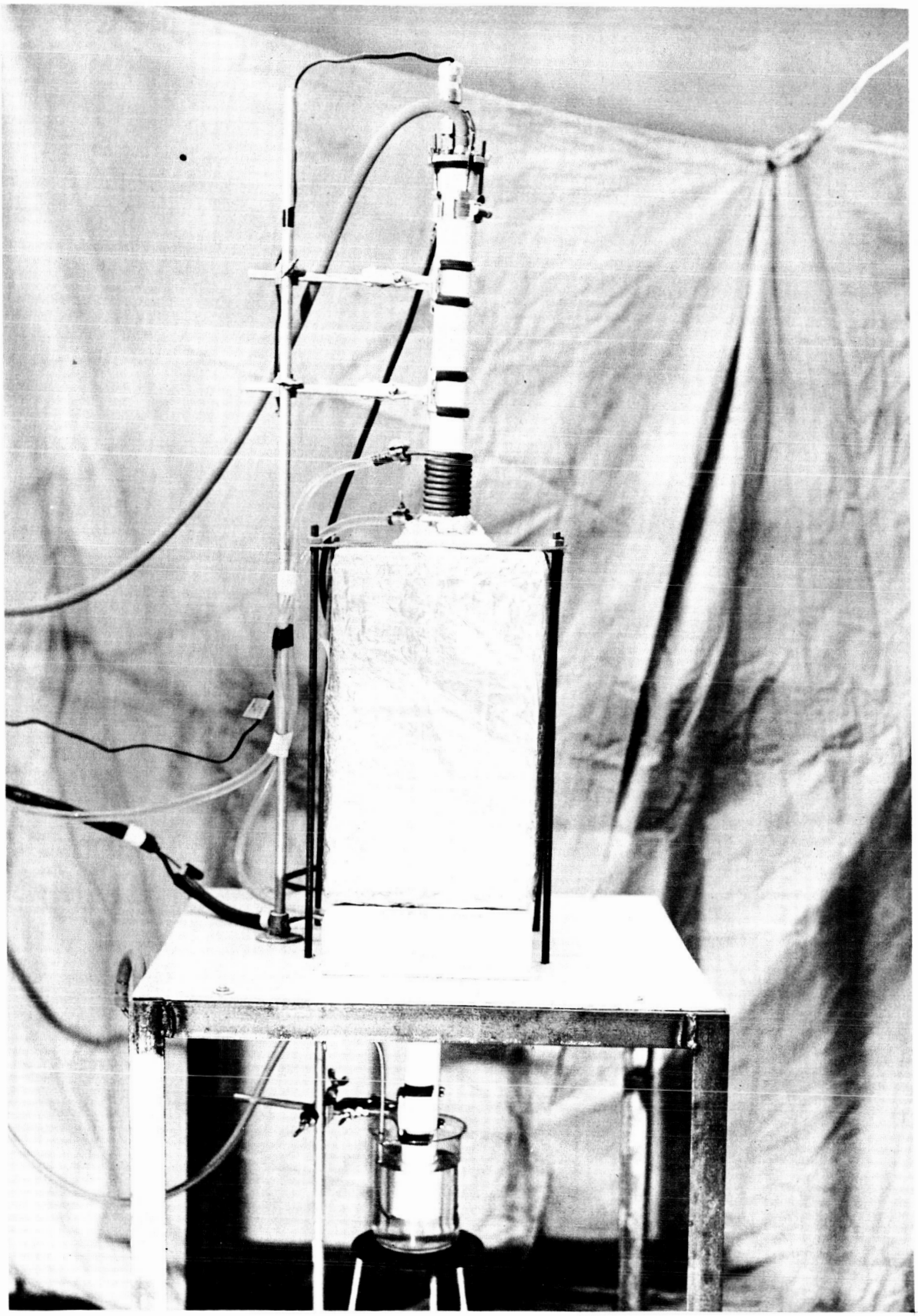

Figure 1.A Quenching Furnace

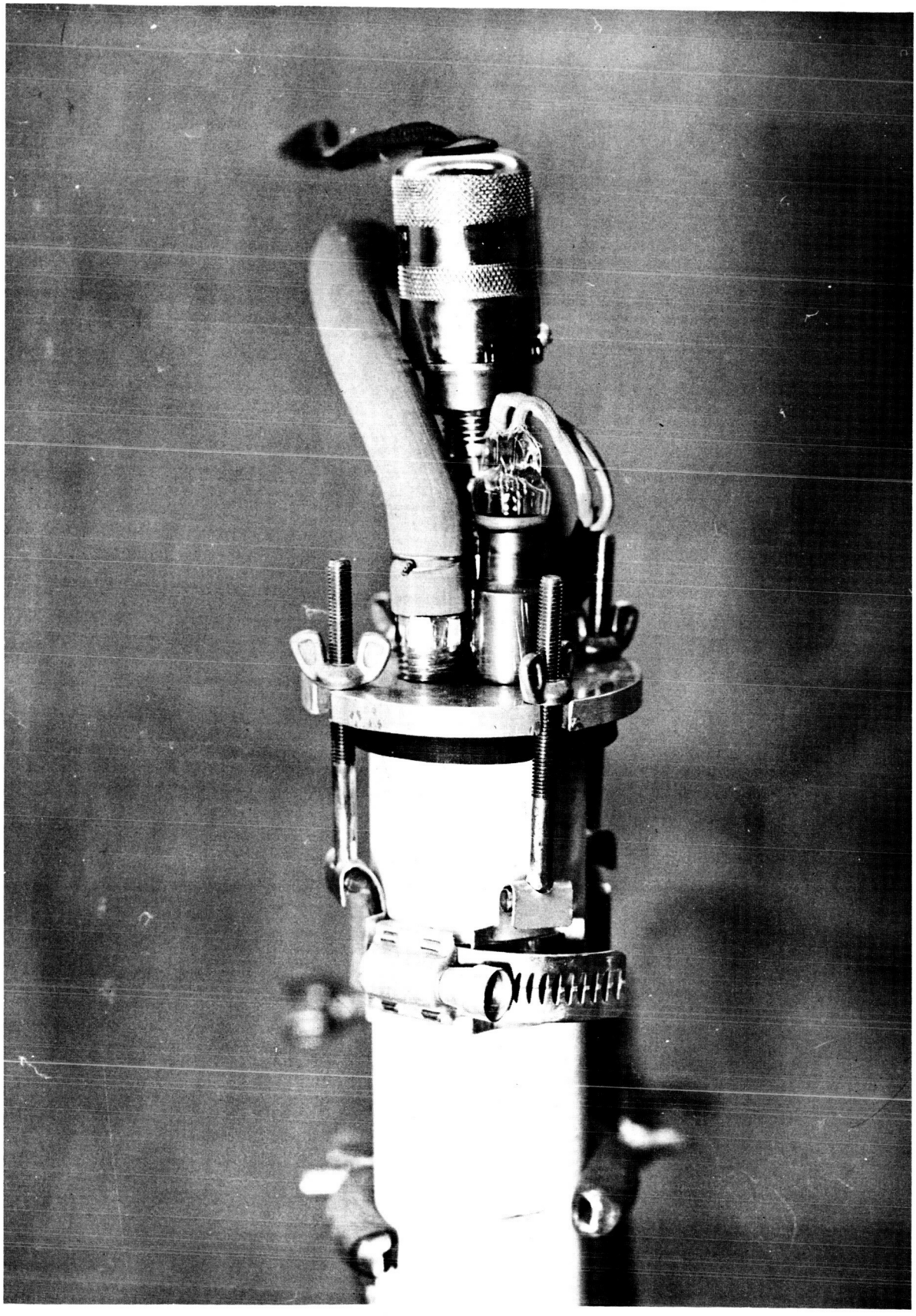

Figure 1.B Current leads to specimen suspension, thermocouple and gas outlet of quenching furnace

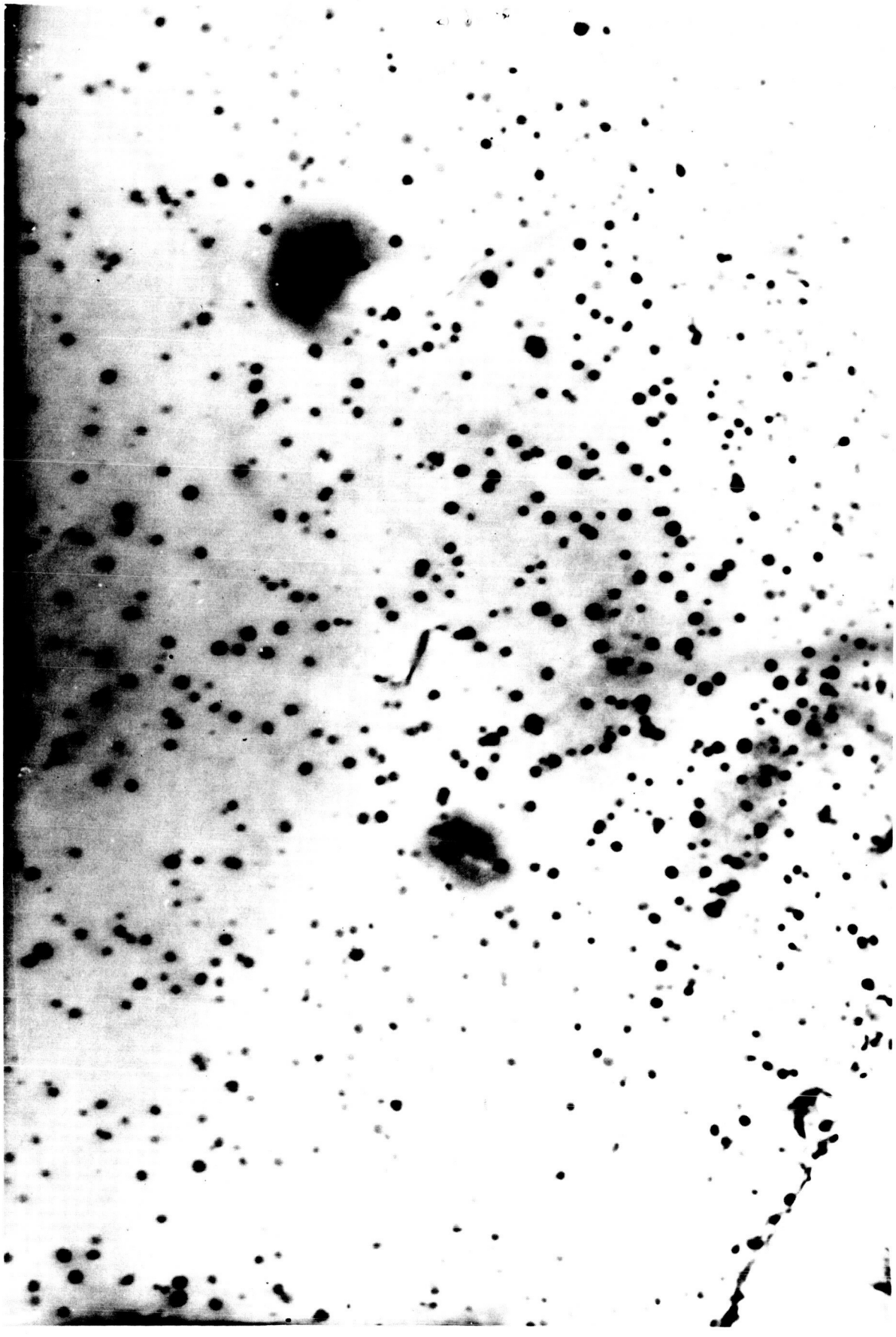

Figure 2 Ni - 1% Cr internally oxidized at 800°C for 20 hours. Mag. = 100,000X