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# ELECTRON METALLOGRAPHIC INVESTIGATIONS OF THE MINOR PHASES OF HEAT-RESISTANT ALLOYS

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## SUMMARY

The progress of the research work on Contract AF-33(616)-3250 during the second quarter of the contract year is described. This work includes: (1) a continuation of the studies of the minor phases of S816 alloy, (2) additional studies of the microstructures of commercial wrought nickel-base alloys and comparison of micro- and macrohardness measurements on these alloys, (3) a continuation of the studies of the microstructures and minor phases of nickel-base alloys containing higher-than-usual amounts of Ti and Al, and, (4) initiation of work to identify the minor phases of a cast nickel-base alloy.

# OBJECTIVE

Various recent metallurgical investigations indicate that the minor phases which precipitate in the heat-resistant alloys during exposure to stresses and high temperatures have a pronounced effect on the metallurgical properties of the alloys. To understand and control better the properties of these alloys it is therefore desirable to have more detailed information on their minor phases. This project has been undertaken in order to employ the highly sensitive techniques of electron diffraction and electron microscopy in identifying the minor phases and in studying their distribution within the alloys, with the overall objective of obtaining such information.

The work of this project falls into two general phases. The first is concerned with the development of various experimental procedures necessary for adapting the techniques of electron diffraction and electron microscopy to the study of the complex heat-resistant alloys. This involves principally the development of polishing, etching, and rinsing procedures for use in preparing surfaces of the various alloys for examination by the electron methods. The general problems associated with this phase of the work have been discussed in WADC Technical Report 54-589 which was prepared under a previous contract. The second phase of the work is concerned with applying these special experimental procedures to studies of typical alloys used in the manufacture of components of jet aircraft engines and similar high-temperature applications. Here close cooperation is maintained with Professor J. W. Freeman and his group in the Department of Chemical and Metallurgical Engineering who are doing metallurgical research in these alloys, and particular attention is given to attempting to correlate variations in the high-temperature properties of the alloys with the variations observed in their minor phases.

## INTRODUCTION

This report describes the experimental work which was carried out during the second quarter of the contract year. This includes additional studies of the minor phases of the S816 alloy; the application of selective etching techniques and extraction replica techniques in studies of the phases of the special 1079 alloy; comparisons of micro- and macrohardness measurements for determination of the precipitation hardening of nickel-base alloys; and preliminary studies of the microstructure of a cast nickel-base alloy, Guy alloy. The first two of these topics represent continuations of work described in Progress Report No. 1 for this contract, while the latter two represent new work undertaken during this quarter. These various topics are discussed individually in the following sections of this report.

## EXPERIMENTAL METHODS

The general procedures for studying the minor phases of alloy systems by electron diffraction and electron microscopy have been described by Heidenreich and his associates,[1,2] and some of the special procedures which have been developed for applying these methods to the heat-resistant alloys have been described in a previous Air Force Technical Report.[3] Briefly, minor phases are identified by electron diffraction patterns obtained by directing the electron beam across surfaces of the alloy specimens which are etched by special techniques so that the minor phases protrude slightly above the matrix metal. Because of the low penetrating power of the electron beam, these patterns arise almost entirely from the protruding minor-phase particles. These patterns are analogous to patterns obtained from fine crystalline powders by x-ray diffraction methods and are interpreted similarly. The size, shape, and distribution of the minor phases are determined by electron microscopic studies which are usually made on the same surfaces used for the diffraction studies. These electron microscopic studies are made through the use of palladium-shadowed collodion replicas. The preparation of these replicas and the interpretation of the resulting micrographs have been described previously.[3]

## STUDIES OF THE MINOR PHASES OF S816 ALLOY

Studies of the minor phases of aged specimens of the S816 alloy have been in progress for some time. In the last progress report[4] an etching procecure was described which produces a preferential attack on the CbC and TiC phases,

permitting them to be distinguished from the complex type of carbides such as $M_6C$ and $M_{23}C_6$ when the etched surfaces are examined by electron microscopy. It was also pointed out that this procedure was advantageous for electron diffraction studies since it removed the large CbC and TiC particles, permitting the electron beam to strike other phases present in the surfaces more readily.

This selective etching procedure has recently been used in conjunction with standard etching procedures in the electron diffraction studies of the minor phases of the S816 alloy. The results which have been obtained to date are summarized in Table I. The phases identified include columbium carbonitride and an $M_{23}C_6$-type complex carbide. In most cases only the Cb(C,N) patterns were obtained by electron diffraction when the surfaces of the specimens were prepared by standard etching procedures. The Cb(C,N) pattern was also the only one obtained by x-ray diffraction methods from residues separated from the various specimens by digestion in alcoholic bromine solutions. The $M_{23}C_6$ phase was identified by electron diffraction from surfaces which were etched with the selective etchant to remove the interference of the very large Cb(C,N) particles.

TABLE I. MINOR PHASES IDENTIFIED IN AGED SPECIMENS OF S816 ALLOY

| Period of Aging (hours) | Temperature of Aging 1200°F | 1400°F | 1600°F |
|---|---|---|---|
| 1 | -- | $M_{23}C_6$<br>Cb(C,N) | $M_{23}C_6$<br>Cb(C,N) |
| 10 | $M_{23}C_6$<br>Cb(C,N) | -- | $M_{23}C_6$<br>Cb(C,N) |
| 20 | -- | $M_{23}C_6$<br>Cb(C,N) | -- |
| 200 | -- | -- | Cb(C,N) |
| 400 | $M_{23}C_6$<br>Cb(C,N) | -- | -- |
| 1000 | -- | $M_{23}C_6$<br>Cb(C,N) | Cb(C,N) |

As indicated in Table I, both the Cb(C,N) and the $M_{23}C_6$ phases have been identified in all specimens except those aged for 200 and 1000 hours at 1600°F. Although repeated examinations have been made of these two specimens, it has

not been possible to date to obtain any evidence of the $M_{23}C_6$ phase from them. On the other hand, very satisfactory patterns of the $M_{23}C_6$ phase have been obtained from the specimens aged 1 and 10 hours at this temperature.

This effect of aging time on the $M_{23}C_6$ phase is very interesting since it is unusual to find a phase forming at a given temperature and then subsequently dissolving at that same temperature. A possible explanation may be offered in terms of competition between the $M_{23}C_6$ and Cb(C,N) phases for the available carbon in the alloy. During the early periods of aging, sufficient carbon may be available to permit both phases to form. The Cb(C,N) phase may be sufficiently more stable than the $M_{23}C_6$ phase at the relatively high temperature of 1600°F so that, as aging progresses, it causes the $M_{23}C_6$ to redissolve by reducing the level of carbon available below that required to maintain the $M_{23}C_6$ phase. This would be consistent with the observation that the Cb(C,N) particles increase very rapidly in size and number during aging at 1600°F. The instability of the $M_{23}C_6$ phase relative to Cb(C,N) at 1600°F was also indicated by the results obtained in the studies of the Inconel-X alloy. The $M_{23}C_6$ and Cb(C,N) were observed in specimens of this alloy aged at 1200° and 1400°F, but only the Cb(C.N) phase was found in specimens aged at 1600°F.

A further investigation of this matter is being made, and attempts are being made to obtain correlations between the metallurgical properties of the S816 alloy and the disappearance of the $M_{23}C_6$ phase at 1600°F.

## MICROHARDNESS MEASUREMENTS ON A NICKEL-BASE ALLOY

In the previous progress report,[4] it was indicated that studies are being made of several commercial nickel-base alloys to attempt to account for differences in their metallurgical properties in terms of differences in their microstructures and compositions. The properties of these alloys are generally considered to depend largely on the formation of an age-hardening precipitate within the matrix grains during aging. Recently, however, questions have been raised as to the contributions to the properties of the alloys by the relatively heavy deposits of carbides which form at the grain boundaries. To obtain some indications of their effect, hardness measurements made by micro techniques, in which the hardness indentations are located entirely within the grains, are being compared with measurements made by the usual macro techniques.

To date, preliminary results have been obtained from a series of specimens of the Udimet alloy. These specimens are selected to represent different degrees of aging as follows: (a) solution-treated 4 hours at 1975°F, ice-brine quenched, (b) solution-treated 4 hours at 1975°F, air-cooled, (c) solution-treated 4 hours at 1975°F, ice-brine quenched, aged 10 hours at 1400°F, (d) solution-treated 4 hours at 1975°F, ice brine quenched, aged 100 hours at 1400°F,

and (e) solution-treated 4 hours at 1975°F, air-cooled, aged 1000 hours at 1400°F.

The hardness of these specimens as measured by the macro and micro methods are listed in Table II. The macrohardness values listed are the averages of values obtained from three widely separated indentations in each specimen. As indicated, the three values for each specimen agreed within ± 3 hardness numbers in every case. The microhardness values are the averages of values from six indentations in each specimen. The average deviation of the individual values from the mean was about ± 20 hardness numbers for all specimens, as indicated. This larger deviation for the micro values is not unreasonable inasmuch as these would be expected to be influenced somewhat by the crystallographic orientations of the individual matrix grains.

TABLE II. HARDNESS OF UDIMET ALLOY SPECIMENS AS DETERMINED BY MACRO AND MICRO METHODS

| Specimen | Macrohardness* | Microhardness** |
|---|---|---|
| a. Ice-brine quenched | 293 ± 3 | 300 ± 20 |
| b. Air-cooled | 347 | 364 |
| c. Aged 10 hours at 1400°F | 401 | 434 |
| d. Aged 100 hours at 1400°F | 419 | 421 |
| e. Aged 1000 hours at 1400°F | 404 | 461 |

*Vickers Hardness Numbers, 50-kg load.

**Vickers Hardness Numbers, 100-gm load.

These micro- and macrohardness values are compared graphically in Fig. 1. Both methods indicate a similar increase in the hardness of the alloy from about 300 for the ice-brine-quenched specimen to about 425 for the specimen aged 100 hours at 1400°F. Electron microscopic studies of these specimens have been completed and show that this increase in hardness is accompanied by a corresponding increase in the amount of the intermetallic $\gamma'$ phase precipitated within the matrix grains of the alloy. As shown by the electron micrographs reproduced in Fig. 2, there are no detectable $\gamma'$ particles in the ice-brine-quenched specimen which has the lowest hardness of the series. In the other specimens the $\gamma'$ particles are readily discernable and increase in size with the aging treatment.

The most interesting factor is the difference in the macro- and microhardness values for the specimen aged 1000 hours. At present, sufficient information is not available to account completely for this difference; however, it

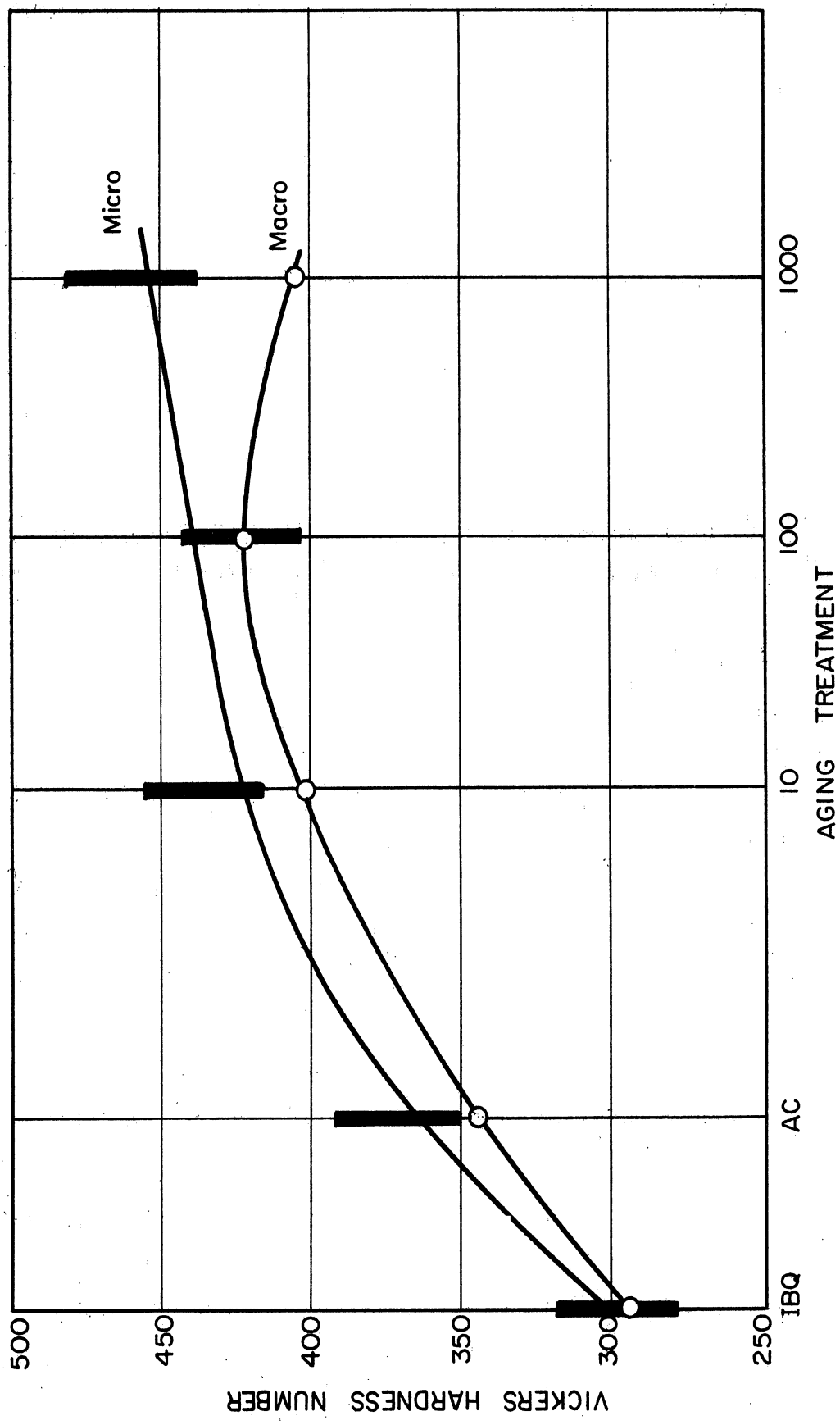
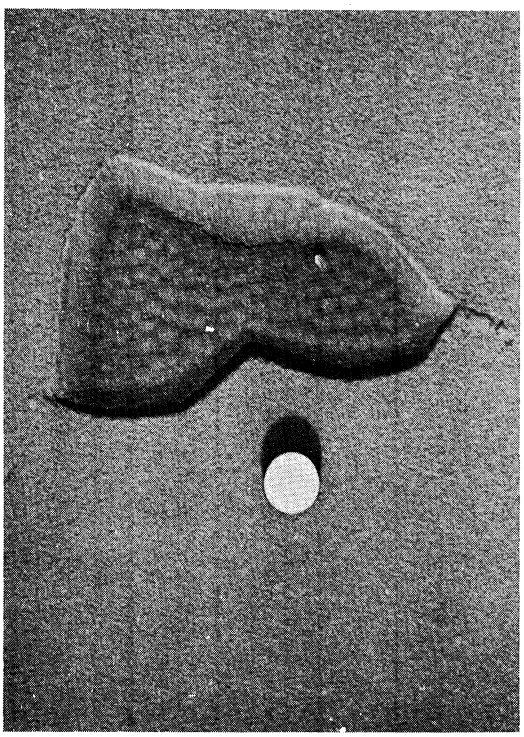
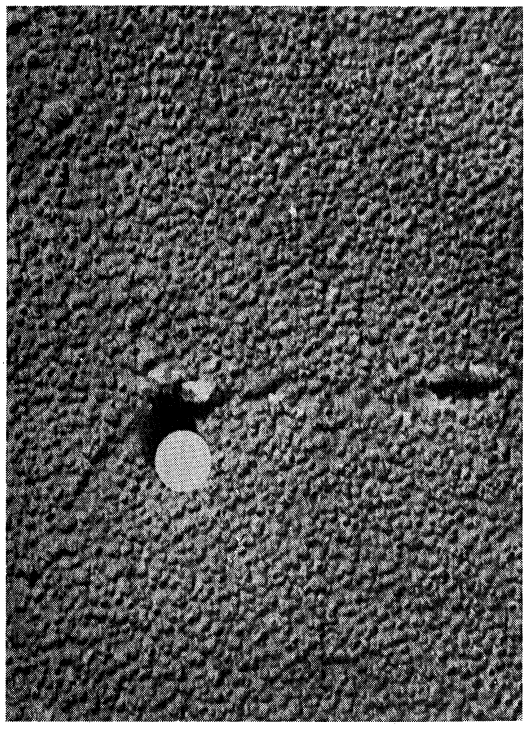



Fig. 1. Comparison of micro- and macrohardness values for Udimet alloy ice-brine quenched and air-cooled from solution treatment at 1975°F, and subsequently aged 10, 100, and 1000 hours at 1400°F.

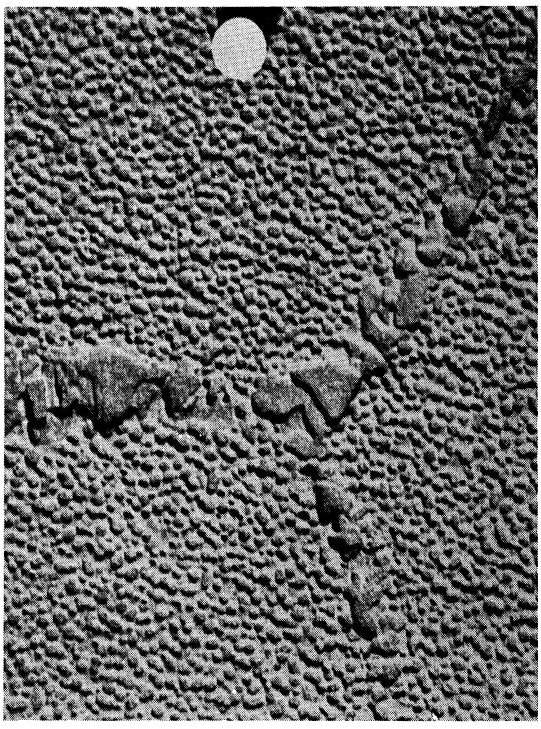


a. Ice-brine quenched

b. Air-cooled



c. Aged 10 hours at 1400°F



d. Aged 100 hours at 1400°F

Fig. 2. Electron micrographs of Udimet alloy. (X 30,000)

does not appear to be due to random fluctuations in the measurements inasmuch as it is considerably greater than the uncertainty in the mean of either set of hardness values. It is possible that there is a softening of the alloy near the grain boundaries due to overaging. This would be reflected in the macrohardness values, but not in the microhardness values, and could account for the observations. If this is the case, a decrease in the rupture strength of the alloy might be expected to accompany the softening of the grain boundaries. It is also possible that surface conditions influence the microhardness measurements sufficiently to cause the observed deviation from the macrohardness values.

These various possibilities are now being studied. Electron microscopic studies are also being made to compare the microstructure of the specimen aged 1000 hours with the microstructures of the other specimens. This should be helpful in explaining the hardness results. In addition, if overaging occurs, as is indicated by the macrohardness measurements, it will be interesting to compare the changes in microstructure accompanying overaging of this alloy with changes previously observed to accompany overaging of the Inconel-X alloy.

## MINOR PHASES OF NICKEL-BASE ALLOYS CONTAINING HIGH PERCENTAGES OF Ti AND Al

Previous studies of a special nickel-base alloy, designated as No. 1079, containing 4% each of Ti and Al (see Table III) showed heavy intergranular precipitation which consisted of angular particles in the as-cast alloy and large cubic particles in specimens solution-treated at 1975°F. These particles were strikingly different from any observed in commercial alloys and were attributed to the higher titanium and aluminum content of the 1079 alloy. It was considered possible that these particles were the $\eta$ phase based on $Ni_3Ti$, rather than the $\gamma'$ phase based on $Ni_3Al$ which is usually found in the commercial alloys; therefore, attempts have been made to obtain information as to their identity.

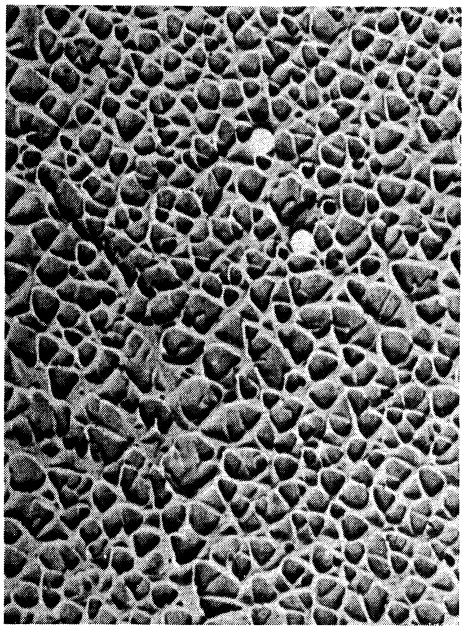
TABLE III. COMPOSITIONS OF SPECIAL NICKEL-BASE ALLOYS

| Alloy | Composition in Weight Percent | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Ni | Cr | Co | Fe | Mo | Ti | Al | Mn | Si | C | B | Cb |
| 1079 | Bal | 19.0 | 16.8 | -- | 3.7 | 4.1 | 4.2 | 0.1 | 0.3 | 0.06 | | |
| 1095 | Bal | 19.5 | 16.5 | -- | 3.4 | 5.8 | 6.0 | 0.1 | 0.3 | 0.07 | | |
| Guy Alloy | Bal | 13.0 | -- | 4.5 | 6.0 | -- | 6.0 | -- | -- | 0.10 | 0.5 | 2.0 |

It has been possible to make use of the extraction replica technique of Fisher[5] to isolate some of the angular particles similar in appearance to those seen in the 1079 alloy from an alloy, designated as 1095, of even higher Ti and Al content (see Table III). These particles were isolated on an electron-microscope replica where electron diffraction patterns were obtained from them directly by the selected area diffraction method. The isolation was accomplished through the use of an electrolytic etch using dilute phosphoric acid. The surface of the specimen was first prepared by the usual techniques to bring the precipitates in relief as shown in Fig. 3a. A collodion replica was then applied and the surface was etched lightly in the phosphoric acid with the replica in place. This etching treatment attacked the matrix metal, freeing the precipitate particles which remained attached to the replica when it was subsequently removed from the surface. The appearance of the isolated particles is shown in Fig. 3b and an electron diffraction pattern obtained from them is reproduced in Fig. 3c. All the reflections of this pattern were found to correspond to the face-centered cubic reflection of the $\gamma'$ phase, and there were no indications of reflections corresponding to the hexagonal $\eta$ phase.

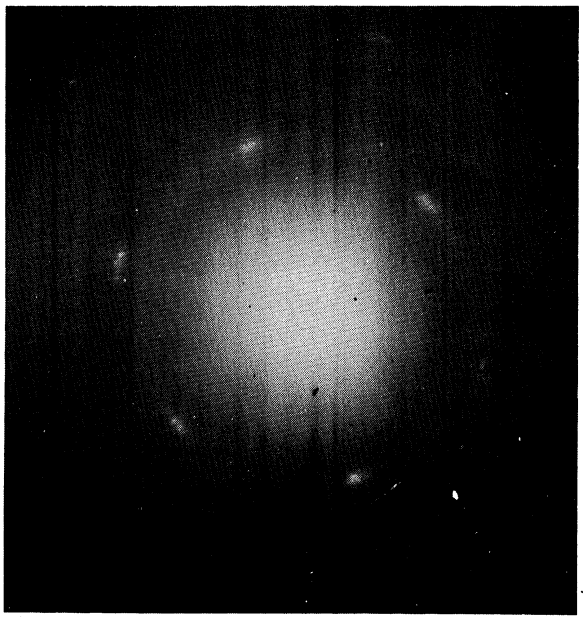
The etching procedure, which has been found to produce a selective attack on the $\gamma'$ phase of the commercial alloys of lower Ti and Al content, was applied to a specimen of the 1079 alloy which had been solution-treated 4 hours at 1975°F and air-cooled to room temperature. The large cubic precipitate particles which occur in this specimen are shown in Fig. 4a, and the attack of the selective etching treatment on these particles is shown in Fig. 4b.

Both of these observations suggest that the intragranular precipitate which forms in the complex, commercial type of nickel-base alloys is the $\gamma'$ and not the $\eta$ phase, even when the alloys contain as much as 4-6% of Ti and Al. On this basis, the microstructures of the four specimens of the 1079 alloy which were discussed in the previous progress report[4] can readily be accounted for. Due to the high Ti and Al content, considerable quantities of the $\gamma'$ phase precipitate in the alloy. In the as-cast condition these particles have the angular shape similar to those shown in the micrograph Fig. 3a. The solution-treating temperature of 1975°F is not sufficient to dissolve this phase completely, but does permit considerable agglomeration of the relatively small particles of the as-cast alloy into larger cubic particles such as those shown in Fig. 4a. When the alloy is ice-brine quenched from solution treatment at 1975°F these are virtually the only particles observed (see Fig. 5c of Progress Report No. 1). When the alloy is air-cooled from solution treatment at 1975°F, however, sufficient aging occurs during cooling to bring about the precipitation of the fine dispersion of much smaller particles in the matrix between the large cubic particles as shown in Fig. 4a. These smaller particles resemble the $\gamma'$ particles which form in the alloys of lower Ti and Al content. Solution treatment at the higher temperature of 2150°F is sufficient to dissolve the $\gamma'$ phase and thus accounts for the absence of the cubic particles in specimens ice-brine quenched from this temperature (see Fig. 5b of Progress Report No. 1).



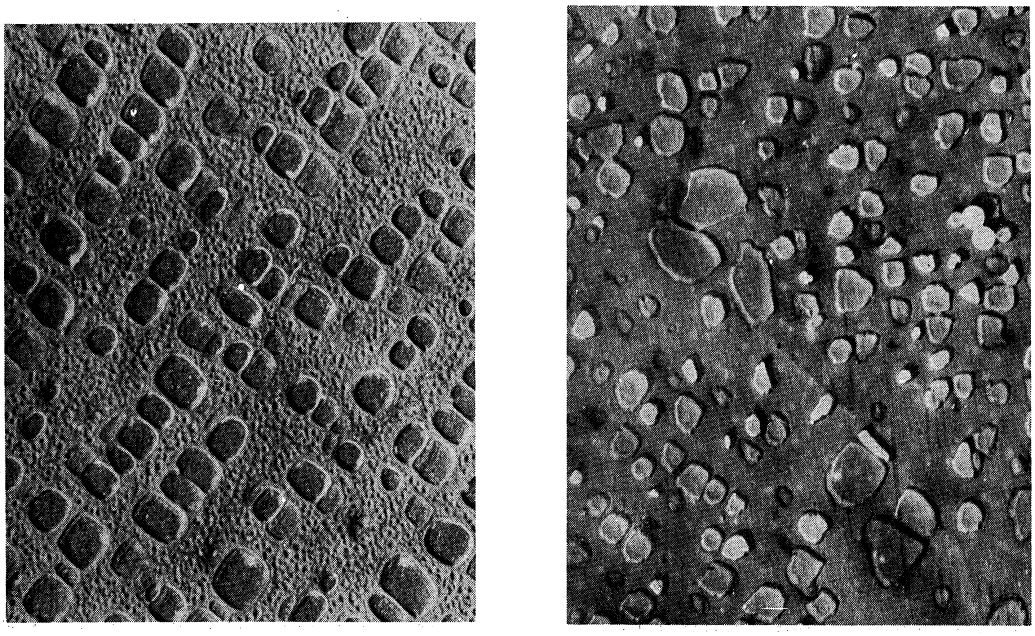
a. Microstructure of specimen (X 12,000)

b. Extracted particles (X 12,000)



c. Electron diffraction pattern

Fig. 3. Application of the extraction replica technique to the special 1095 nickel-base alloy.



a. Regular etch b Selective etch

Fig. 4. Application of the $\gamma'$ selective etching treatment to the special 1079 nickel-base alloy. (X 12,000)

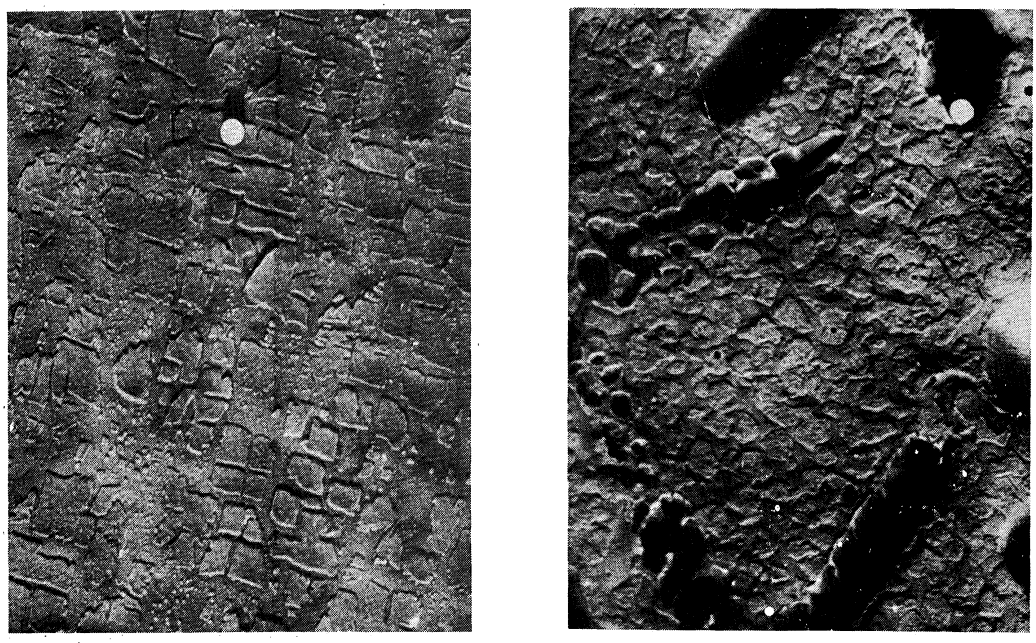
These results are of interest in that they suggest that the results obtained by Nordheim and Grant[6] and Taylor and Floyd,[7] indicating the presence of the $\eta$ phase in alloys of simpler compositions but having high Ti and Al contents, may not carry over to complex alloys of the types used for commercial applications. Attempts are being made to obtain diffraction patterns of the particles in the 1079 alloy using the extraction replica techniques described here for the 1095 alloy.

## MICROSTRUCTURE AND MINOR PHASES OF CAST NICKEL-BASE ALLOYS

Cast nickel-base alloys are widely used in production of turbine blades and other precision parts for aircraft engines. At present considerable interest is attached to understanding the dependence of the metallurgical properties of these alloys on the different variables of the melting and casting processes. Of particular interest is the fact that alloys produced by vacuum-melting techniques usually have much higher strengths than alloys produced by air-melting methods, even though no significant compositional differences can be detected. Considerable metallurgical research is being done on this problem in the Department of Chemical and Metallurgical Engineering, and microstructure and minor-phase studies are being carried out on this project to correlate with these metallurgical studies.

The alloy being used in this work is known commercially as Guy alloy, whose aim composition is given in Table III. In contrast to the wrought nickel-base alloys, such as Udimet, M-252, Inconel-X, and Waspalloy, which have been studied so far, this alloy contains no titanium, but contains considerable boron and relatively high amounts of columbium. Specimens of vacuum- and air-melted heats of this alloy are being examined by electron and x-ray diffraction and electron microscopy in the as-cast condition and after rupture testing at 1500°F to see if structural differences can be observed which will account for the higher strength of the vacuum-melted heats. Preliminary results have been obtained in the various phases of this work.

In the as-cast specimens the electron micrographs show a discontinuous phase in the form of large particles of more or less cubic shape and irregular size which are distributed in a continuous phase. These two phases present a "flagstone" pattern in the micrographs. It appears that the discontinuous phase comprises at least half of the volume of the alloy. In the rupture-tested specimens the discontinuous phase is considerably distorted from its cubic form but appears to be present in approximately the same amount as in the as-cast specimens. In addition to these two phases, precipitates appearing as long stringers and accumulations of individual rounded particles are also present in both the as-cast and the rupture-tested specimens. In optical micrographs these present an interesting "Chinese script" pattern. Electron micrographs from as-cast and rupture-tested specimens of a vacuum heat are reproduced in Fig. 5 and show these various microstructural features. To date it has not been possible to detect significant differences in the microstructures of vacuum-melted and air-melted specimens.



a. As cast b. Rupture tested

Fig. 5. Electron micrographs of vacuum-melted Guy alloy specimen. (X 12,000)

Attempts are being made to identify the various phases of this alloy. X-ray diffraction patterns have been obtained from residues separated, by digestion with a 10% solution of bromine in methanol, from the as-cast and rupture-tested specimens of both the air- and vacuum-melted heats. The same pattern was obtained from all specimens. The interplanar spacings and relative intensities of the diffraction lines of this pattern are listed in Table IV. It has not yet been possible to arrive at an entirely satisfactory identification of the materials producing this pattern. As indicated in Table IV, several of the strongest lines agree very well with CbC lines, and one or two others agree reasonably well with those of the austenitic matrix phase. The remainder of the lines do not fit patterns of any of the common carbide or nitride phases for which diffraction data are available. Because of the high

TABLE IV. X-RAY AND ELECTRON DIFFRACTION PATTERNS FROM GUY ALLOY SPECIMENS

| X-Ray Pattern | | E. D. Pattern | | CbC Pattern | | Matrix Pattern | | $M_2B$* Pattern |
|---|---|---|---|---|---|---|---|---|
| d | I | d | I | d | I | d | I | d |
| | | | | | | | | 3.55-3.94 |
| | | | | | | | | 3.25-3.33 |
| 3.09 | m | 3.09-3.30 | w | | | | | |
| 2.87 | w | 2.81-2.87 | w | | | | | |
| 2.56 | s | 2.71-2.76 | w | 2.55 | s | | | 2.51-2.79 |
| 2.46 | m | | | | | | | |
| 2.20 | w | 2.21-2.27 | w | 2.21 | s | | | 2.14-2.37 |
| 2.10 | s | 2.05-2.10 | w | | | 2.08 | s | 1.98-2.20 |
| 2.03 | wm | | | | | | | 1.83-2.03 |
| 1.98 | s | | | | | | | |
| 1.82 | w | 1.83-1.86 | w | | | | | 1.78-1.97 |
| | | | | | | 1.79 | s | 1.63-1.80 |
| | | | | | | | | 1.59-1.77 |
| 1.56 | m | 1.56-1.61 | w | 1.56 | s | | | |
| 1.54 | vw | | | | | | | |
| 1.40 | vw | | | | | | | 1.37-1.52 |
| 1.36 | vw | | | | | | | 1.33-1.47 |
| 1.34 | ms | | | 1.33 | s | | | 1.28-1.42 |
| 1.27 | ms | 1.27-1.29 | w | 1.27 | m | 1.27 | m | 1.26-1.40 |
| 1.24 | vw | | | | | | | 1.20-1.33 |
| | | 1.09 | w | | | | | |

*The d values are calculated from unit cell dimensions reported for different metal borides.[8] Intensities are not known.

boron content of the alloy, it is possible that the precipitates are borides or borocarbides. If this is the case, identification will be difficult because of the lack of published x-ray diffraction data on such compounds. Included in Table IV is a listing of interplanar spacings calculated from cell dimensions reported by Kiessling[8] for several metal borides having the general formula $M_2B$. The x-ray data agree only fairly well with these values, and since intensity data are not available the identification is very uncertain.

Electron diffraction patterns have also been obtained from etched surfaces of the different specimens, using reflection diffraction techniques. The patterns obtained to date have been generally of rather poor quality, consisting of weak, diffuse spots and having rather heavy backgrounds. Because of their spotty character, accurate measurement has not been possible; however, it appears that the patterns are essentially the same for all specimens. The approximate values of the interplanar spacings of the patterns are included in Table IV. It will be noted that they correspond fairly well with the values obtained by x-ray diffraction and with the calculated $M_2B$ values.

Both the x-ray and electron diffraction patterns probably were produced by the "Chinese script" precipitate phases, since the electron micrographs show these to be the most resistant to attack (see Fig. 5b). The continuous and discontinuous phases giving rise to the "flagstone" pattern in the electron micrographs appear to be intermetallic phases, and from the composition of the alloy might be expected to be the $\gamma$ and $\gamma'$ phases. It is interesting that the continuous phase appears to be in relief with respect to the discontinuous phase in the surfaces studied by electron microscopy (Fig. 5). These surfaces were etched electrolytically with a reagent consisting of 5 ml of hydrofluoric acid (48%), 10 ml of glycerol, and 85 ml of ethyl alcohol. With other nickel-base alloys, this reagent has been found to leave the $\gamma'$ phase in relief with respect to the $\gamma$ phase.[9] If this is the case with the present alloy, then it would appear that the continuous, or matrix, phase of this alloy is the $\gamma'$ phase and that the $\gamma$ phase occurs as a precipitate. This is the opposite of what has been observed for the alloys studied previously.

Considerable additional work is indicated for this alloy. Attempts are being made to obtain better standard diffraction data to use in identifying the phases producing the x-ray and electron diffraction patterns. Attempts are also being made to obtain better electron diffraction patterns by improving the etching conditions. The principal difficulty, however, appears to result from the fact that the specimens have very large grains so that the electron beam strikes particles having only a few different orientations. Provisions are being made to move the specimen relative to the beam during exposure to try to improve this situation. It is also proposed to make spectrographic analyses of the extracted minor phases to determine the principal elements in them and to look for differences in their compositions depending on the method of melting the alloy.

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