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

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## SUMMARY

The progress of the experimental work on Contract No. AF 33(616)-3250 during the last half of the contract year is described. This work includes: (1) further examination of the carbide reactions in S816 alloy, (2) extension of microstructural examination of commercial wrought nickel-base alloys, and (3) initiation of studies of the ω-phase in Ti-8Cr and Ti-13Mo alloys.

## 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

A manuscript entitled "Submicroscopic Precipitates in Commercial Nickel-Base Alloys," which was prepared for publication in a technical journal, was submitted in lieu of a Progress Report for the second quarter of the contract year. This report therefore deals principally with the experimental work carried out during the last quarter of the contract year, but also includes work done during the second quarter which was not incorporated in the manuscript. Described here are: further studies of the carbides in S816 alloy aged at 1600°F; identifications of the $\gamma'$ phase in Inconel X, Udimet, Waspalloy, and M-252 alloy by electron diffraction from extraction replicas; continuation of the investigations of the relation of microstructure and hardness in the Udimet alloy; and the development of polishing and etching techniques for electron metallographic studies of Ti-8Cr and Ti-13Mo alloys. The first three of these topics are continuations of work described in Progress Reports Nos. 1 and 2 for this contract, while the third topic represents new work undertaken since the last report. Work on the Guy alloy has been temporarily discontinued, inasmuch as members of the Department of Chemical and Metallurgical Engineering are now carrying out studies of this material. The following sections of the report describe the results obtained to date in each of these investigations.

## 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 so etched by special techniques that the minor phases protrude slightly from 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

Previous progress reports[4,5] have described studies of the minor phases of S816 alloy specimens aged at 1200°, 1400°, and 1600°F. Electron microscopic examinations have shown that these specimens characteristically contain three types of precipitate particles: very large, slightly angular particles which are randomly distributed through the alloy and are present even in solution-treated specimens prior to aging; smaller particles which form in the grain boundaries during aging; and fine particles which generally form within the grains after the grain-boundary precipitation is well advanced. From the results of electron diffraction and electron microscopic studies on specimens aged at 1200° and 1400°F, it was concluded that the large angular particles were Cb(C,N) and that the other precipitates were a complex carbide of the $M_{23}C_6$ type. Recent results from the specimens aged at 1600°F have raised some questions concerning this latter point, for although the $M_{23}C_6$ phase has been detected in specimens aged 1 and 10 hours, it has not been detected in specimens aged 200 and 1000 hours which show all types of precipitates.

In order to resolve these questions, it was decided to attempt to employ the extraction replica method of Fisher.[6] In this method the surface is initially etched to bring the precipitate particles into relief, an appropriate replica film is applied and the surface is etched a second time with the replica in place. This frees the particles from the surface but leaves many of them embedded in the replica film with the same orientation and relative location as in the alloy. When the replica is transferred to the electron microscope the particles can be viewed directly, and electron diffraction patterns can be obtained from selected known groups of them by the selected-area electron diffraction technique.

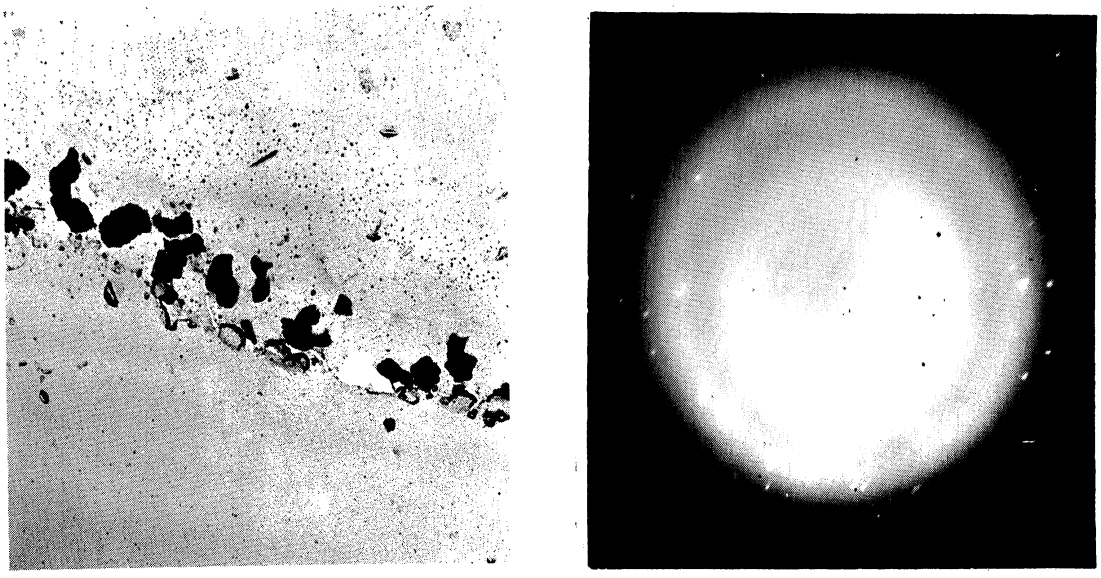
To date examinations have been made of the alloy specimens aged 1, 10, 200, and 1000 hours at 1600°F. The initial preparation of these specimens was accomplished by electrolytic polishing in a solution of 10 parts perchloric acid (70%) and 90 parts glacial acetic acid, followed by electrolytic etching in a solution of 12 parts phosphoric acid (85%), 41 parts nitric acid (70%), and 47 parts sulfuric acid (96%). Carbon replica films deposited on the etched surfaces by evaporation in a vacuum,[7] were used because of their resistance to chemical attack, and the second etching treatment was carried out

electrolytically using one of the following reagents:

a. 1 part nitric acid (70%) and 3 parts hydrofluoric acid (48%)
b. 48% hydrofluoric acid.

Reagent a was chosen for this purpose because previous results[5] indicated that it dissolves the Cb(C,N) phase without dissolving the $M_{23}C_6$ phase. The use of this reagent should therefore permit the $M_{23}C_6$ particles to be picked up on the replicas, but not the Cb(C,N) particles. It was hoped that reagent b would not attack either of these phases and would thus permit particles of both to be picked up on the replicas.

Where reagent a was used in the preparation of the specimens aged 1 and 10 hours, the precipitate particles from the grain boundaries were picked up by the replica, but not the large blocky particles. A typical electron micrograph of these grain boundary particles is reproduced in Fig. 1a. The electron diffraction patterns obtained from these particles by the selected-area diffraction technique consisted of a large number of diffraction spots as shown in Fig. 1b. The interplanar spacings corresponding to these spots are listed in Table I. Due to the character of these patterns and difficulties in obtaining accurate calibration of the instrument, measurements of the highest accuracy were not possible; nonetheless, the agreement with the reported values for the $M_{23}C_6$ carbide is quite satisfactory. Similar results were obtained when reagent b was used in preparing the specimens except that some of the large angular particles were also picked up on the replicas. Generally, no satisfactory diffraction patterns were obtained from these particles, probably because they were too large to transmit the electron beam.



a. Electron micrograph (X1500). b. Electron diffraction pattern.

Fig. 1. Results obtained by the extraction replica technique; S816 alloy specimen aged 10 hours at 1600°F.

TABLE I

INTERPLANAR SPACINGS FROM A SELECTED-AREA DIFFRACTION PATTERN OF GRAIN-BOUNDARY MATERIAL FROM A SPECIMEN OF S816 ALLOY AGED 10 HOURS AT 1600°F

| Observed Data | Standard Data for $M_{23}C_6$ |
|---|---|
| 3.67 Å | 3.76 Å |
| 2.96 | 3.21 |
| 2.64 | 2.66 |
| 2.42 | 2.38 |
| 2.16 | 2.17 |
| 2.04 | 2.04 |
| 1.87 | 1.88 |
| 1.82 | 1.79 |
| 1.76 | 1.77 |
| 1.71 | 1.68 |
| | 1.62 |
| 1.58 | 1.60 |
| 1.52 | 1.53 |
| 1.47 | 1.49 |
| 1.44 | 1.42 |
| 1.37 | 1.38 |

Using these same techniques and procedures, it has been possible to date to isolate only the large Cb(C,N) particles from the specimens aged 200 and 1000 hours. This may be interpreted to indicate that the grain-boundary and intragranular precipitates in these specimens differ from the corresponding precipitates in the specimens aged 1 and 10 hours. The nature of this difference cannot be deduced from the data presently available, but may involve either composition or crystallographic structure. On the other hand, the inability to extract these precipitates from the 1600°F specimens may be dependent on particle size. In the case of the grain-boundary precipitate, the particles may have grown too big by the additional aging to be freed by the etching treatments used, while a sufficient attack may have been produced on the very small intragranular particles to dissolve them or prevent them from remaining attached to the replicas.

Further work is in progress to resolve these questions. This will involve attempts to develop etches which will permit the precipitates to be removed from the specimens aged 200 and 1000 hours at 1600°F for the extraction replica studies, as well as application of standard electron diffraction, electron microscopic, and x-ray diffraction methods. There is particular interest in these results, for Rowe and Freeman[8] have recently suggested that decreases in the rupture properties of the alloy accompanying overheating to 1800°F result from replacement of other precipitated phases by Cb(C,N). Some indica-

tion of this transformation may be obtained from the present studies on the specimens aged at 1600°F. It is also proposed to make studies of the minor phases of the overheated specimens as part of the future work on this alloy.

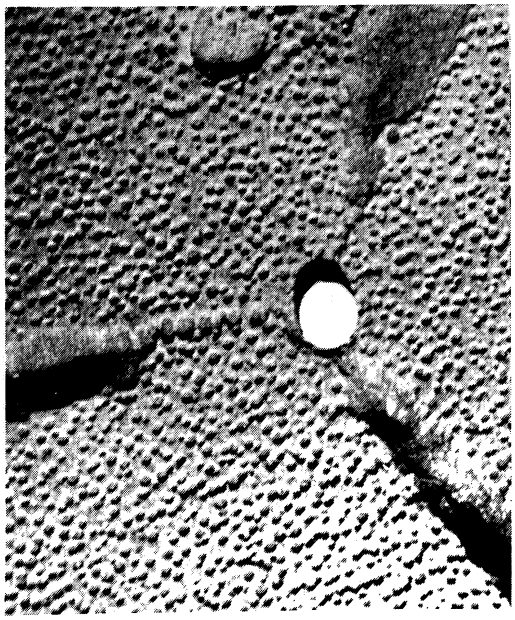
## IDENTIFICATION OF THE $\gamma'$ PHASE OF THE NICKEL-BASE ALLOYS

In the work to date, the identification of the intragranular pricipitate in commercial nickel-base alloys as the intermetallic $\gamma'$ phase has been based largely on its etching characteristics[9] and on x-ray diffraction results obtained from the Inconel-X alloy.[3] More direct identification of these particles has long been desirable, particularly in connection with the studies of the newer alloys such as Udimet, M-252, and Waspalloy. To accomplish this, the extraction replica techniques described above have been employed so that the particles could be isolated from the matrix and studied directly by electron diffraction and electron microscopy. The procedures used were generally the same as those described above for the S816 alloy, except that the second etching treatment was carried out electrolytically in a 15% phosphoric acid solution.

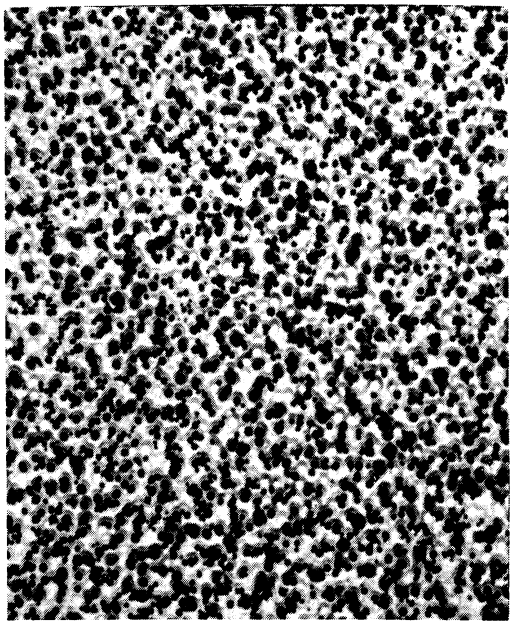
This method has been applied to date to specimens of Udimet, Waspalloy, and M-252 alloys aged 100 hours at 1400°F, and to specimens of Inconel-X alloy aged 1000 hours at 1400°F and 10 hours at 1600°F. The results have confirmed the identification of the intragranular particles of all these specimens as the $\gamma'$ phase and have also provided evidence that this phase has an ordered structure based on a face-centered cubic lattice in all these alloys.

As an illustration of these results, a conventional electron micrograph from the M-252 alloy specimen aged 100-hours at 1400°F is reproduced in Fig. 2, together with an electron micrograph and an electron diffraction pattern from the intragranular particles isolated from this specimen by the extraction replica procedure. The similarity in the shape and distribution of the particles in the two micrographs is quite convincing, and the fact that the particles appear slightly smaller in the extraction replica is undoubtedly due to particle dissolution during the extraction etching process. The electron diffraction pattern consists primarily of a few single-crystal diffraction spots corresponding to the usual face-centered cubic reflections having all even or all odd Miller indices. In addition, careful examination of the original photographic plates revealed several much weaker spots at positions corresponding to the remaining cubic reflections and indicating an ordered structure. The Miller indices and interplanar spacings calculated for all these reflections are listed in Table II. As shown, there is very good agreement with values calculated from data on the size of the unit cell of the $\gamma'$ phase reported by Nordheim and Grant.[10]

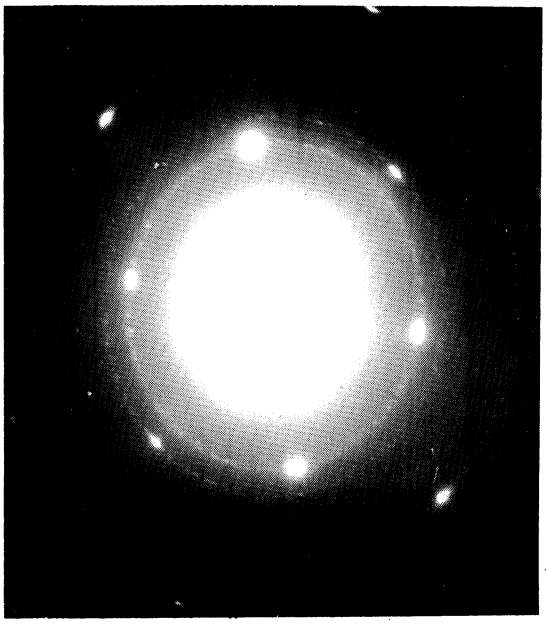
Since the extracted particles have the same orientation in the



a. Regular electron micrograph (X30,000).



b. Micrograph from extraction replica (X30,000).



c. Selected-area diffraction pattern from particles in Fig. 2b.

Fig. 2. Application of extraction replica techniques to M-252 alloy aged 100 hours at 1400°F.

TABLE II

ELECTRON DIFFRACTION DATA FROM $\gamma'$ PHASE
OF M-252 ALLOY AGED 100 HOURS AT 1400°F

| $d_{hkL}$ Values from Extraction Replicas | Miller Indices | Calculated Pattern for Ordered $Ni_3(Ti,Al)$ |
|---|---|---|
| 3.56 Å | 100 | 3.56 Å vw |
| 2.52 | 110 | 2.52 vw |
| 2.06 | 111 | 2.06 s |
| 1.78 | 200 | 1.78 s |
| 1.59 | 210 | 1.59 vw |
| 1.45 | 211 | 1.45 vw |
| 1.26 | 220 | 1.26 s |
| 1.19 | 300/221 | 1.19 vw |
| | 310 | 1.13 vw |
| 1.07 | 311 | 1.07 s |
| 1.03 | 222 | 1.03 s |
| $a_o$ = 3.56 Å | | $a_o$ = 3.56 Å |

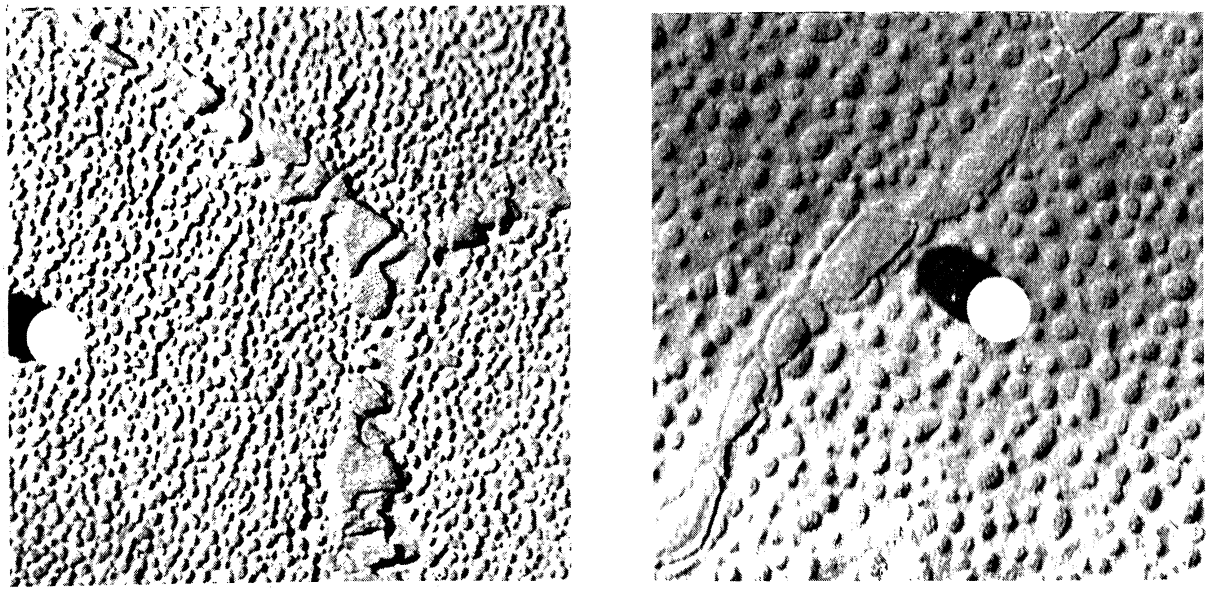
replica as in the metal surface, some indication of the degree of preferred orientation of the particles in the alloys can be obtained from comparison of the character of the electron diffraction patterns obtained from the replicas. A high degree of preferred orientation will result in patterns consisting of a few strong spots, while a random orientation will result in patterns of more or less spotty rings. The results obtained to date indicate a high degree of preferred orientation in the Inconel-X, Udimet, and M-252 specimens, but very little in the Waspalloy specimen. This is a rather interesting observation and will receive further investigation to see if there is any relationship between the orientation of the $\gamma'$ particles and the compositions and properties of the alloys.

## MICROSTRUCTURE AND HARDNESS OF UDIMET ALLOY

In the previous report, studies of the hardness of Udimet alloy specimens aged at 1400°F were described which indicated that maximum hardness was reached with about 100 hours of aging at this temperature, and a decrease from a Vickers hardness value of 419 to a value of 404 occurred with further aging to 1000 hours. Differences between macro- and microhardness values were also noted which were indicative of a softening of the grain boundaries of the alloy with long periods of aging. Electron micrographs showing the microstruc-

tures of the specimens aged for periods up to 100 hours were presented, but micrographs of the specimen aged 1000 hours were not available for correlation with the over-aging effects.

The electron microscopic studies of this latter specimen have now been completed and a representative micrograph is reproduced in Fig. 3 together with a micrograph from the specimen aged 100 hours. Comparison of these two micrographs shows that increasing the period of aging from 100 to 1000 hours produces a considerable increase in the size and separation of the $\gamma'$ particles, and a marked decrease in the number of these particles per unit volume. There is also a noticeable increase in the size of the carbide particles in the grain boundaries, accompanied by the formation of a layer of $\gamma'$ phase surrounding these particles.



a. Aged 100 hours. b. Aged 1000 hours.

Fig. 3. Electron micrographs from specimens of Udimet alloy aged at 1400°F (X30,000).

In considering the metallurgical implications of these observations, it is of particular interest to note that the microhardness values determined from indentations made within the grains of the specimens were the same for the specimen aged 1000 hours as for the specimen aged 100 hours. This indicates that the considerable change in the size and distribution of the $\gamma'$ particles within the matrix grains had very little effect on the hardness of the grains. The decrease observed in the macrohardness of the alloy therefore appears to be associated with the microstructural changes which occurred at the grain boundaries. This is of particular interest, for in independent studies Freeman and Decker have observed layers of the $\gamma'$ phase at the grain boundaries of rupture-tested specimens of the alloy, and have obtained some evidence that the formation of these layers is most pronounced in specimens having the lowest rupture strengths.

Similar studies have been started on a series of Udimet alloy specimens aged at 1600°F. Macrohardness measurements have been completed on these specimens and show maximum hardness to be reached with between 1 and 10 hours of aging, and a considerable decrease which accompanies further aging to 100 hours. The observed values are plotted in Fig. 4. Only preliminary microhardness measurements have been completed to date, and these have been rather unsatisfactory. In general, they have been much lower than the macrohardness values and varied over a wide range for each specimen. Considerable additional work to refine these measurements is therefore necessary.

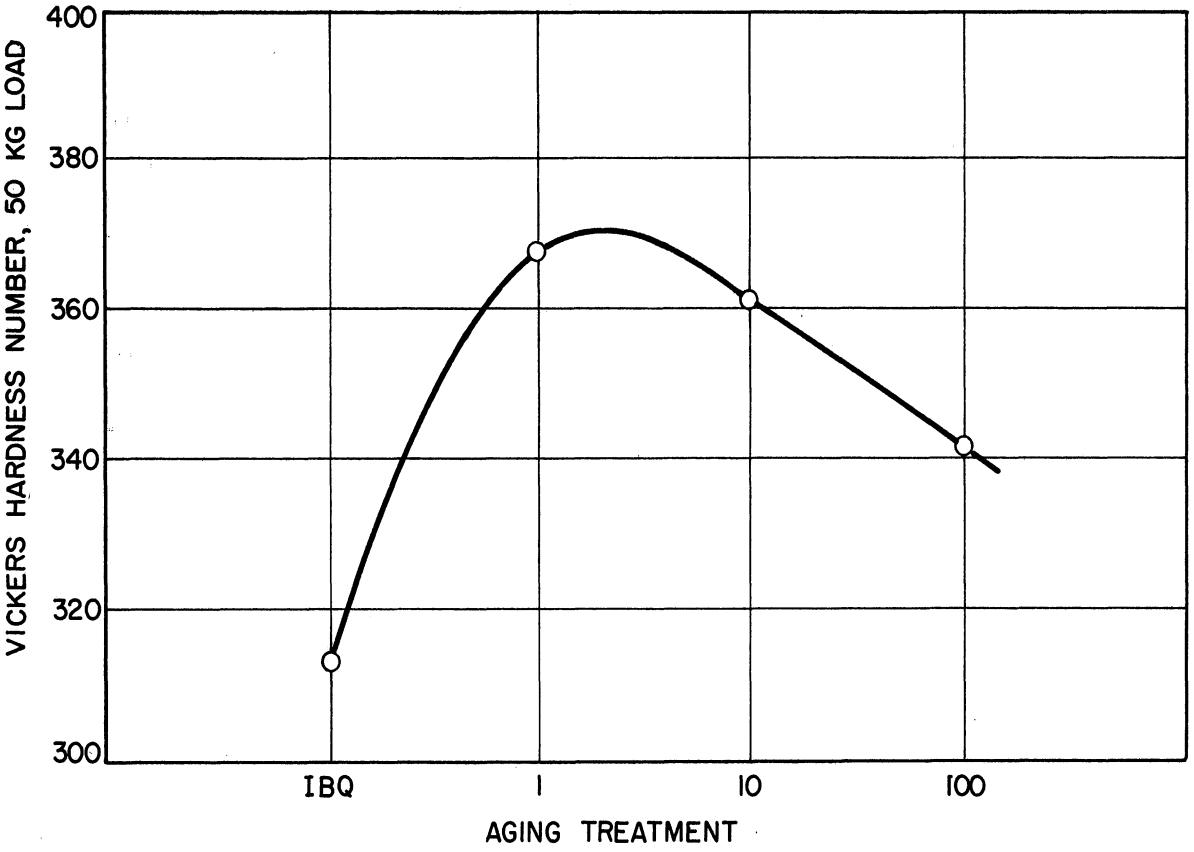



Fig. 4. Hardness values for Udimet alloy specimens, ice-brine quenched from solution treatment at 1975°F, and subsequently aged 1, 10, and 100 hours at 1600°F.

Electron microscopic studies of the microstructures of these specimens are also in progress. Figure 5 shows the microstructure of the specimen aged 100 hours at 1600°F. The $\gamma'$ particles in this specimen are very much larger than those produced by 1000 hours of aging at 1400°F, and the development of the layers of the $\gamma'$ phase at the grain boundaries is also much more extensive. These studies are being continued to obtain an overall picture of the effect of aging at various temperatures on the microstructure and hardness of the alloy.

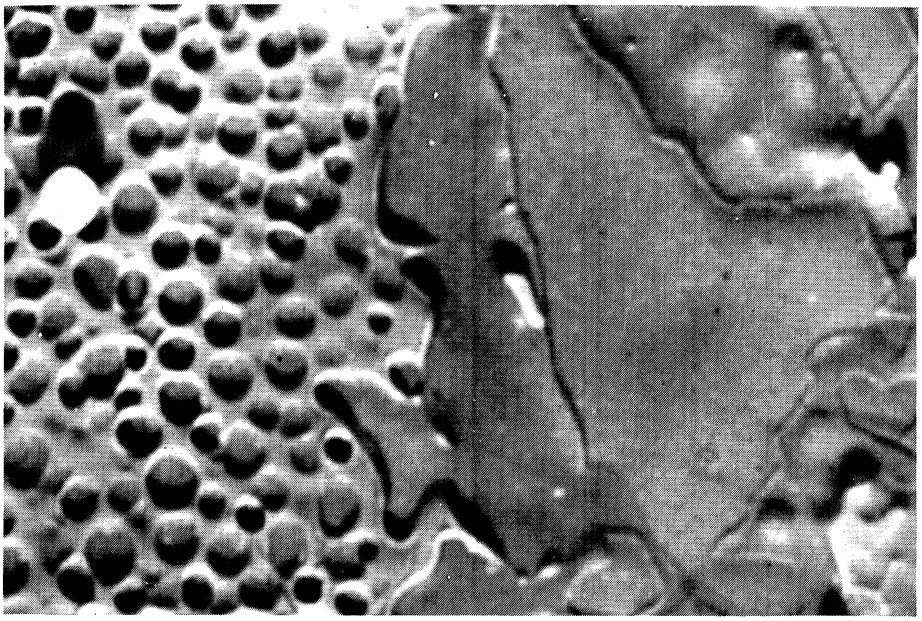


Fig. 5. Microstructure of Udimet alloy specimen aged 100 hours at 1600°F (X30,000).

## INVESTIGATION OF THE ω PHASE IN Ti ALLOYS

Improvement of metallurgical properties by precipitation of a second phase in an alloy is not confined to the nickel-base alloys discussed above. Among the other important alloys exhibiting this phenomenon are certain of those based on titanium, in which hardening is produced by the precipitation of the low-temperature α-titanium (hexagonal) within the matrix of the high temperature β form (body-centered cubic). In some alloys this precipitation process involves the formation of a metastable transition structure which is referred to as the ω phase. The exact crystallographic structure of this phase is somewhat doubtful and has been reported to be orthorhombic with $a_o$ = 6.2, $b_o$ = 6.5, $c_o$ = 13.5 Å,[11] or to be face-centered cubic with $a_o$ = 9.8 Å.[12] The orientation of the ω particles relative to the β matrix is also uncertain.

At the request of the Metallurgy Research Branch of the Aeronautical Research Laboratory, WADC, attempts have been made to employ electron microscope and diffraction techniques to obtain information that might clarify these points. For these studies, specimens of Ti-8Cr and Ti-13Mo alloys were obtained by WADC from Dr. W. Rostoker and associates at the Armour Research Foundation. These specimens were reportedly solution-treated at 900°C, water-quenched, and aged to produce maximum content of the ω phase with minimum α content (8 hours at 400°C for the Ti-8Cr alloy and 4.2 hours at 400°C for the Ti-13Mo alloy).

As is generally the case whenever studies of a new type of alloy are

undertaken, considerable difficulty has been encountered in developing procedures which will produce the high quality of polishing and etching required for electron diffraction and microscopy. Both electrolytic and mechanical polishing methods have been tried, and more than 20 different electrolytes for the electrolytic polishing have been investigated. These have included the electrolytes recommended by Tajima[13] and Osadchuk[14] for titanium and its alloys, and various modifications of these. An equal number of etching reagents, both immersion and electrolytic, have also been tried. Again these have included the various reagents recommended for etching titanium alloys for optical microscopy, and various modifications thereof.

Due to the high resistance of the Ti-8Cr and Ti-13Mo alloys to electrochemical attack, electrolytic polishing results were generally not very satisfactory, and when mechanical polishing was employed, extreme difficulty was encountered in removing the polishing powders to obtain surfaces clean enough for electron diffraction. The most satisfactory overall results were obtained by mechanically polishing with 3/0 emery, then with Linde "A" alumina on a wax lap, and following this with a light electrolytic polish in one of the reagents listed in Table III to remove mechanically worked metal and the polishing alumina from the surfaces. Most of the etching reagents tried produced pitting and staining of the surfaces; however, it was possible to obtain satisfactorily etched surfaces, particularly of the Ti-8Cr alloy specimen, by electrolytic etching using low current (0.1 to 0.2 amp) in polishing solution 1 of Table III.

Two of the best electron micrographs which have been obtained from the alloys are reproduced in Fig. 6. From these it appears that both alloys contain a very fine dispersion of precipitate particles, and there is some suggestion that these are in the form of rods or platelets. Their "diameter" or "thickness," as the case may be, appears to be of the order of 400 Å. It is felt that these particles are the ω phase, for their size and distribution as indicated by the electron micrographs are consistent with the conclusions of Rostoker[11] based on x-ray diffraction and metallurgical evidence, and with the hardening accompanying aging.

The electron diffraction results to date have been less satisfactory in that it has not been possible to obtain patterns which can be definitely identified as arising from the ω phase. The principal difficulty apparently involves surface contamination in the polishing and etching operations. Because of the extremely small size of the ω particles, the merest traces of contaminants deposited on the surfaces would be sufficient to screen them from the electron beam. In some cases, patterns consisting of an array of single crystal spots corresponding to the cubic β phase have been obtained. This is encouraging since it indicates that sufficiently clean surfaces have been prepared. Slight modifications of the etching techniques may lead to patterns of the ω phase.

In continuing this work it is proposed to attempt the refining of

polishing and etching procedures developed so far to improve the reproducibility of the electron microscopic results and to permit electron diffraction patterns to be obtained of the ω phase. The possibility of employing extraction replica techniques is also being considered.

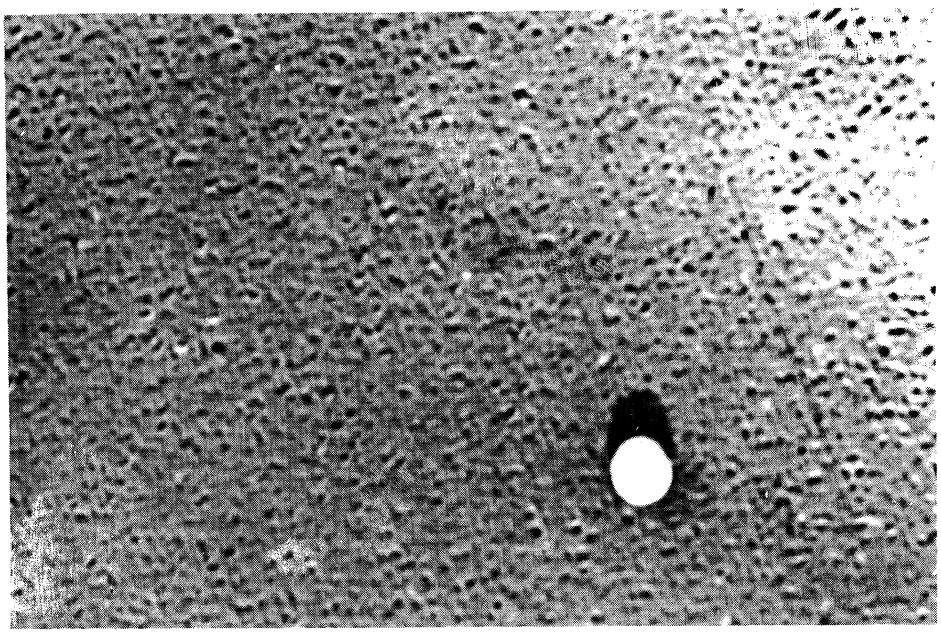
TABLE III

POLISHING SOLUTIONS FOR TITANIUM ALLOYS

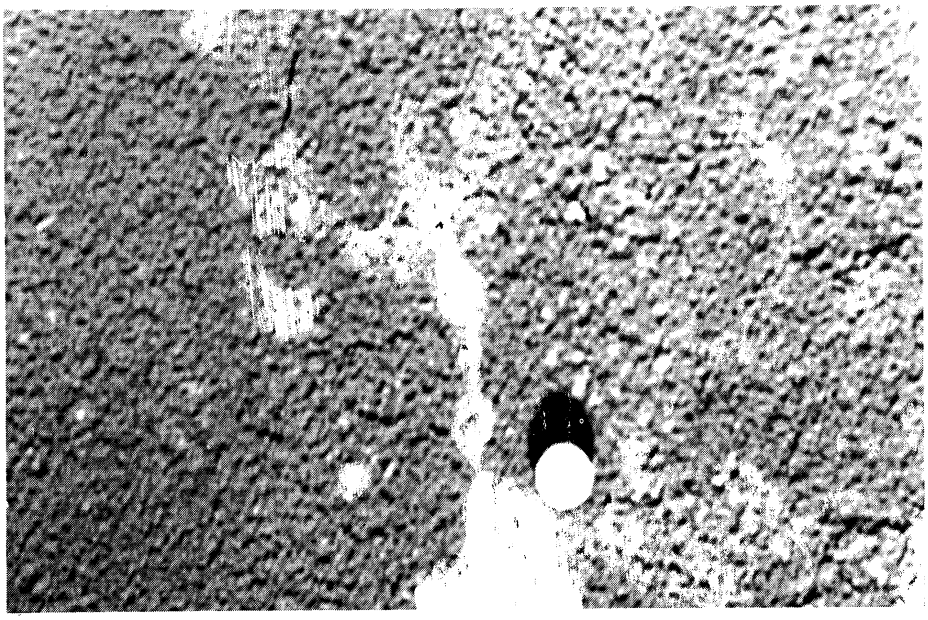
(1) 77 ml acetic acid (glacial)
15 ml chromic acid (43%)
8 ml hydrofluoric acid (48%)
Must be fresh for best results.
Temperature about 10°C; current, 3-5 amp.
(Best for Ti-13Mo alloy)

(2) 100 ml solution (1)
6 ml perchloric acid (70%)
Use as above.

(3) 37 ml nitric acid (tech., conc.)
37 ml lactic acid (85%)
18.5 ml hydrochloric acid (37%)
7.5 ml hydrofluoric acid (48%)
Temperature 60-80°, immersion.
(Best for Ti-8Cr alloy)



a. Ti-8Cr alloy



b. Ti-13Mo alloy.

Fig. 6. Electron micrographs from the titanium-base alloys (X30,000).

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