

ENGINEERING RESEARCH INSTITUTE
UNIVERSITY OF MICHIGAN
ANN ARBOR

QUARTERLY PROGRESS REPORT NO. 1
Period 15 January to 15 April 1952

DEVELOPMENT OF PROCEDURES FOR THE IDENTIFICATION OF
MINOR PHASES IN HEAT-RESISTANT ALLOYS BY ELECTRON DIFFRACTION

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Project 2020

WRIGHT AIR DEVELOPMENT CENTER, U. S. AIR FORCE
CONTRACT NO. AF-33(616)-23, E.O. No. R463 Br-1

15 April 1952

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SUMMARY

The adaptation of electron diffraction methods to the identification of minor constituents in heat-resistant alloys is under investigation.

Studies are being carried out to select suitable polishing, etching, and rinsing procedures for preparing specimens of 16-25-6, S816, Inconel-X and low-carbon N-155 alloys for electron diffraction studies.

Results to the present include the identification of a CbC-CbN phase in N-155 and S816 alloys and of an M_6C phase in 16-25-6 alloy. Strong indications of an $M_{23}C_6$ carbide and of a face-centered cubic phase with a unit cell edge of 6.0 Å have also been obtained for N-155 alloy.

Further studies, directed toward the correlation of the occurrence of the minor phases with the metallurgical treatments and microstructures of the alloy specimens, are in progress.

INTRODUCTION

One of the present limitations to the construction of more efficient and more powerful jet engines for aircraft use is imposed by the lack of suitable heat-resistant alloys from which to manufacture the high-temperature components of the engines. Studies of the alloys currently being used for this purpose indicate that the variability of their properties at high temperatures is related to the precipitation of small quantities of minor-phase materials such as carbides and nitrides within the matrix of the alloys. In forming, these precipitates alter the concentrations of elements in the alloy matrix, thereby influencing such properties of the alloys as their resistance to oxidation and corrosion. In addition, the presence of the particles in the matrix grains and at the grain boundaries strongly affect the creep resistance and rupture strength of the alloys. The evaluation of these effects is complicated due to the fact that the quantity, and perhaps the nature, of the precipitates changes as the alloys are subjected to mechanical stresses or high temperatures for prolonged periods. It is evident, therefore, that the ability to identify these minor-phase materials would be of great value in understanding the properties of the heat-resistant alloys now in use and also in developing the metallurgical principles necessary for compounding alloys having more desirable properties.

It is difficult to effect this identification, however, and the currently available methods, such as microscopic examination of etched or stained specimens, x-ray diffraction methods, and chemical and spectroscopic analyses, are generally limited in their effectiveness by one or more of the following factors: (1) inability to give a precise identification of the structure of the minor phases or of the chemical elements involved; (2) inability to detect small quantities of minor-phase materials in the presence of the major phase; and (3) necessity of purifying and separating minor phases from the matrix of the alloys. An improved method which is not subject to these limitations would be of great value.

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Heidenreich and Sturkey^{1,2,3} have recently introduced a method which looks very promising in this respect. In this method the minor-phase materials are identified by electron diffraction patterns obtained from polished and etched alloy samples. The method has been applied with remarkable success to several copper, aluminum, and magnesium alloys and to some ordinary steels. In some cases precipitate particles have been detected and identified which were so small that they could not be seen by ordinary microscopic examination. In addition, preliminary experiments have been carried out at the University of Michigan, prior to starting the present project, which indicate that the method may be suitably adapted to the study of heat-resistant alloys having Fe, Cr, Ni, and Co as principal constituents. The method offers several advantages over those listed above: first, it affords a more positive and discriminating identification of minor phases than is obtained by optical examination of etched or stained alloy specimens; second, it is extremely sensitive, and materials present to an extent of only 0.01 to 0.1 per cent of the total alloy composition may be detected; third, a separation of the minor phases from the matrix phase of the alloy is not required, so that phases may be identified which might otherwise be destroyed in carrying out a separation; and fourth, the distribution of the minor-phase particles in the alloy may be determined by examining the samples from which the diffraction patterns were obtained by electron or optical microscopy.

It is the object of the present investigation to develop the necessary techniques for applying this method to the study of the minor phases of heat-resistant alloys. This report contains a brief description of the experimental procedures required by the method and of the alloys which have been selected for study, together with a discussion of the progress of the work during the first quarter of the contract year.

This work is being carried out in close cooperation with Dr. J. W. Freeman and his coworkers in the Department of Chemical and Metallurgical Engineering of the University, who are currently studying the metallurgy of heat-resistant alloys.

EXPERIMENTAL PROCEDURES

Choice of Alloy Samples

The alloys which have been selected for this investigation are among those which have been studied extensively by the members of the metallurgical engineering staff of the University and include (1) low-carbon N-155, (2) 16-25-6, (3) S816, and (4) Inconel-X alloys. For each alloy a number of different samples have been chosen, each having received different

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thermal or mechanical treatment as indicated in Table I. The selection of

Table I. Heat Treatment of Alloy Samples

<u>Sample</u>	<u>Heat Treatment</u>
Low-Carbon N-155 Alloy:*	
1a-A	Solution-treated for 1 hr at 2200°F; water-quenched.
1-B	Solution-treated for 10 hrs at 2200°F; water-quenched.
1-C	Solution-treated for 1 hr at 2200°F; water-quenched; aged 10 hrs at 1200°F; air-cooled.
1-D	Solution-treated for 10 hrs at 2200°F; water-quenched; aged 1 hr at 1400°F; air-cooled.
1a-E	Solution-treated for 1 hr at 2200°F; water-quenched; aged 24 hrs at 1400°F; air-cooled.
1-F	Solution-treated for 1 hr at 2200°F; water-quenched; aged 24 hrs at 1400°F; air-cooled.
1-G	Solution-treated for 10 hrs at 2200°F; water-quenched; aged 1000 hrs at 1400°F; air-cooled.
1-H	Solution-treated for 10 hrs at 2200°F; water-quenched; aged 1000 hrs at 1600°F; air-cooled.
1a-J	Solution-treated for 2 hrs at 2050°F; water-quenched; reduced 25% at 1200°F; rupture-tested 319 hrs at 1350°F under 33,000 psi.
1-K	Solution-treated for 1 hr at 2200°F; water-quenched; reduced 40% at 2000°F.
* Samples 1a are taken from a different heat than samples 1.	
16-25-6 Alloy:	
2-A	Solution-treated for 2 hrs at 2050°F; water-quenched.
2-B	Solution-treated for 2 hrs at 2050°F; water-quenched; aged 1 hr at 1400°F; air-cooled.

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Table I (cont)

Sample	Heat Treatment
16-25-6 Alloy: (cont)	
2-C	Solution-treated for 2 hrs at 2050°F; water-quenched; aged 1000 hrs at 1400°F; air-cooled.
2-D	Solution-treated for 2 hrs at 2050°F; water-quenched; aged 10 hrs at 1600°F; air-cooled.
2-E	Solution-treated for 2 hrs at 2050°F; water-quenched; aged 1000 hrs at 1600°F; air-cooled.
2-F	Solution-treated for 2 hrs at 2050°F; water-quenched; reduced 15-20% at 1500°F; aged 6 hrs at 1275°F; air-cooled; creep-tested 11,873 hrs at 1300°F at 12,500 psi; tensile-tested at room temperature.
S816 Alloy:	
3-A	Solution-treated for 1 hr at 2300°F; water-quenched.
3-B	Solution-treated for 1 hr at 2300°F; water-quenched; aged 16 hrs at 1400°F; air-cooled.
3-C	Solution-treated for 1 hr at 2300°F; water-quenched; aged 1000 hrs at 1400°F; air-cooled.
3-D	Solution-treated for 1 hr at 2300°F; water-quenched; aged 1000 hrs at 1600°F; air-cooled.
Inconel-X Alloy:	
4-A	Solution-treated for 4 hrs at 2050°F; water-quenched.
4-B	Solution-treated for 4 hrs at 2050°F; water-quenched; aged 1000 hrs at 1400°F; air-cooled.

these samples has been based on metallurgical studies of the high-temperature behavior of the alloys. For example, it has been observed⁴ that the precipitation of minor phases in low-carbon N-155 alloy aged at 1400°F proceeds more or less in three stages: (1) a precipitate is observed to be present

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in the "solution treated", unaged alloy; (2) during the first 24-30 hours of aging, precipitation is confined mainly to the grain boundaries; and (3) thereafter precipitate particles are observed to form within the grains, and this process is accompanied by a decrease in the size of the unit cell of the matrix phase. In 16-25-6 alloy aged at 1400°F, there is an initial decrease in the matrix cell size during the first hour of aging. The cell size then remains constant until after about 10 hours of aging, whereupon it decreases again and reaches a constant value after about 1000 hours.

In addition, some recent, unpublished microscopic and x-ray studies carried out at this University suggest the possibility of sigma phase occurring in samples of N-155 and 16-25-6 alloys, such as 1a-J and 2-F, which have been mechanically treated at moderately high temperatures. The results of these several investigations suggest the precipitation of two or more minor phases in each of these alloys, although the only study which has been reported to date⁵ has detected only a single precipitate in each alloy: CbN in N-155 and an M_6C -type carbide in 16-25-6. For this investigation, samples of each of these alloys have been chosen to permit a study of these and other questions which have been raised by various metallurgical investigations. Similar considerations have been used in picking samples of S816 and Inconel-X alloys, and it is probable that more samples of these alloys will be added as this work progresses.

Preparation of Alloy Samples

The overall procedures used to prepare the samples for electron diffraction studies are essentially the same as were used by Heidenreich and Sturkey¹. The samples are first abraded with metallographic emery papers through 3/0 or 4/0 grades and are then electrolytically polished to remove traces of worked metal and to produce surfaces which are reasonably smooth and flat. These surfaces are then etched, using suitable reagents and conditions, so that the matrix phase is preferentially attacked and the minor-phase particles are left protruding in slight relief. Following this the samples are rinsed with reagents to remove residues from the etching and polishing baths as well as oxidation products of the etching treatment, and to leave the surfaces dry, grease-free, and ready for use in obtaining electron diffraction patterns of the protruding minor-phase particles.

It appears necessary to carry out the final step in polishing the surfaces by electrolytic means rather than mechanically on cloth-covered polishing wheels, as is commonly done for metallographic examinations. The mechanical polishing distorts and works the metal surfaces to such an extent that it becomes difficult to obtain reliable diffraction patterns even after etching the surfaces strongly.

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The etching process may be carried out electrolytically or with reagents which attack the surfaces chemically. Finding reagents suitable for etching the highly inert and easily passivated alloys being studied in this investigation is considerably more difficult than for the more active and somewhat simpler alloys which were studied by Heidenreich and Sturkey. It appears that electrolytic etching is more convenient for use here, although the matter is complicated by the fact that samples of the same alloy which have received different heat treatments often respond differently to the same etching treatment.

The use of proper rinsing procedures following the etching of the surfaces is an extremely important factor in determining the success of the method. The rinses must remove completely all traces of the etching solutions as well as oxidation products from the surfaces of the alloy samples and, in addition, must leave the surface dry and grease-free. For the relatively inert alloys being used in this investigation the problem of rinsing is fairly simple when the etching reagents do not leave insoluble residues on the samples, in which case the following sequence appears to be satisfactory:

First rinse: Grease-free distilled water

Second rinse: 50% methanol, 50% acetone

Third rinse: Benzene

After the third rinse, the etched surfaces are held vertical and allowed to drain and air-dry. It is necessary that the reagents used in making the rinses be freshly distilled from a well-cleaned still, for even analytical grade solvents contain sufficient dissolved grease to leave a grease film when evaporated from the etched surfaces which interferes with obtaining good diffraction patterns. It should be noted that a number of the etching reagents which have been tried produce visible precipitates on the alloys which are not removed by the above series of rinses. If these etchants are to be used, more effective rinses will have to be developed.

Examination of Alloy Samples

Electron Diffraction. The electron diffraction patterns of the minor-phase materials are obtained with an RCA Type EMD electron diffraction unit. This instrument produces a fine, intense beam of 50-kv electrons and is equipped with a versatile set of electron optics which makes it suited for obtaining high-resolution patterns of the type needed in this work.

The alloy samples are oriented in the diffraction camera so that the electron beam strikes the etched surfaces at a grazing angle. Due to the very low penetrating power of the electron beam, the patterns which are

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obtained in this way arise primarily from protuberances on the surfaces. Thus diffraction patterns can be obtained from minor-phase particles which are etched in slight relief from the matrix grains. In addition, the scattering power of materials for electrons is very great (about 10^6 times that of the same materials for x-rays), and satisfactory patterns can be obtained from a very minute quantity of a properly exposed minor-phase material.

The electron diffraction patterns of the minor phases obtained under these circumstances are analogous to the x-ray diffraction patterns which would be produced by a microcrystalline powder of the same material and consist of a series of concentric rings. The identification of the materials producing the patterns is accomplished by calculating the interplanar spacings (d_{hkl} -values) of the crystallographic planes producing the rings and then comparing these values and the relative intensities of the rings with similar data for known compounds of the elements comprising the alloys. Some of the data necessary for making this comparison are available from compilations published by the American Society for Testing Materials and also from the investigations of x-ray workers on metallurgical problems. New phases whose patterns have not been reported can usually be characterized by their crystallographic cell size and type, although their chemical compositions cannot be determined from their diffraction patterns alone.

Microscopic Examination. After the electron diffraction patterns have been recorded, the etched surfaces may be examined by electron or optical microscopy to correlate the diffraction results with the phase distribution and general microstructure of the samples. Electron microscopy is most useful in this respect, for its high magnifications reveal details not visible otherwise, and it also indicates the particles which are protruding from the surface and which are therefore in a condition to contribute to the production of electron diffraction patterns.

Electron micrographs of the surfaces are made using shadow-cast collodion replicas which are prepared in the following manner: A drop of a dilute solution of collodion in amyl acetate is placed on the etched surface of the sample being studied. When the solvent evaporates, a thin film of collodion remains which conforms to the contours of the surface. A small amount of water is caused to condense on the sample by breathing heavily upon it. This water penetrates the film and aids in subsequent separation of the film from the metal. A small disc of 200 mesh screen is then placed on the film, and a piece of Scotch tape is pressed firmly over the disc and is worked in with the fingers so that it adheres to the film as closely as possible out to the edges of the disc. One end of the tape is then lifted, and the tape is carefully peeled from the surface. The collodion film and the disc adhere to the tape and are stripped from the surface. With sufficient care it is possible to remove the entire collodion film, including that which is beneath the gauze disc. The tape is then bent double with a crease beneath the wire gauze, and the gauze is lifted from it with a pair of

tweezers, using extreme care not to wrinkle or damage the collodion film which covers the gauze. The surface of the film which is away from the gauze is a negative replica of the metal surface and reproduces features of the surface which are as small as 10 Å. The contrast of the replica is improved by evaporating a thin film of a metal such as gold or uranium on it at a low angle. The metal deposits only on those portions of the replica which are not shaded from the metal source due to the contours of the replica. When an electron beam is passed through this shadow-cast replica, the image recorded on a photographic plate will look somewhat like an aerial photograph taken when the sun is low in the sky. It must be remembered that when prints are made from these photographic plates, the shadows will appear light rather than dark and that features which appear as protrusions on the replica are actually depressions in the original metal surface.

RESULTS

Previous Studies

Prior to undertaking the present investigation a number of preliminary experiments were carried out to investigate the applicability of the method to the study of heat-resistant alloys. These experiments were carried out with samples 1a-A, 1-F, and 1a-J of N-155 alloy and samples 2-B and 2-D of 16-25-6 alloy (Table I). The electrolytic polishing was carried out in a solution consisting of 100 g of CrO_3 dissolved in 150 ml of concentrated sulfuric acid, 600 ml of concentrated phosphoric acid, and 120 ml of water, using current densities of about 1 ampere per square inch. Samples were electrolytically etched in the polishing solution or in a solution of 45 g of citric acid, 30 g of potassium iodide, and 6 ml of concentrated hydrochloric acid dissolved in 100 ml of water, at current densities of 0.2 to 0.8 ampere per square inch for periods ranging up to 4 minutes. The samples were then rinsed with the series of rinses described above. With these procedures the following results were obtained.

A minor phase which is either CbC or CbN was identified in each of the N-155 samples when they were etched lightly in either the polishing solution or the citric acid, potassium iodide reagent. It was not possible to distinguish unequivocally between these two compounds from the electron diffraction patterns because they have identical crystal structures with nearly the same unit cell size. In addition to the diffraction rings produced by the CbC-CbN minor phase, the patterns from these samples usually contained rings from the matrix phase of the alloy. A typical pattern is reproduced in Fig. 1a.

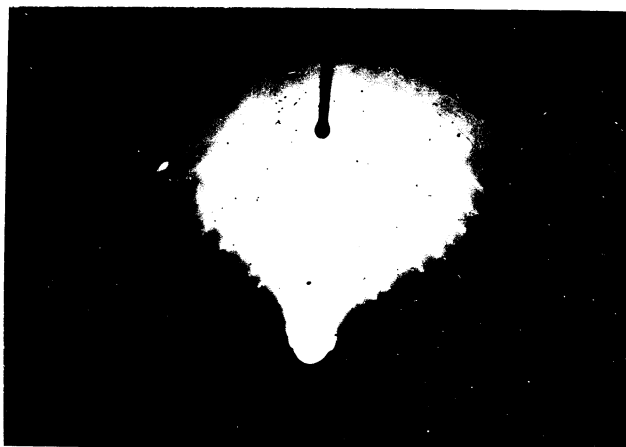


Fig. 1. Cross-section of stem of *Phytolacca* (10x).

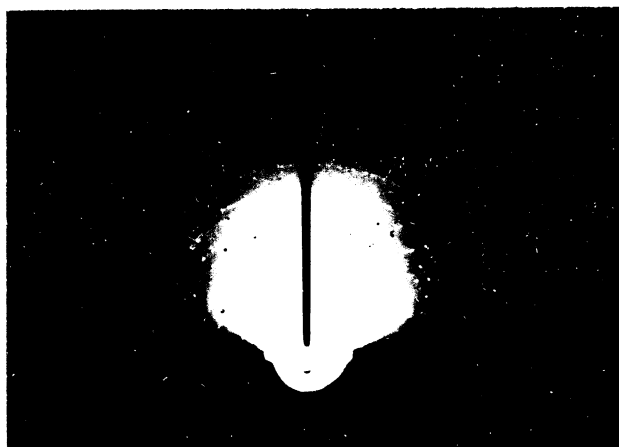


Fig. 2. Cross-section of stem of *Phytolacca* (10x) with an artificial vascular cylinder.

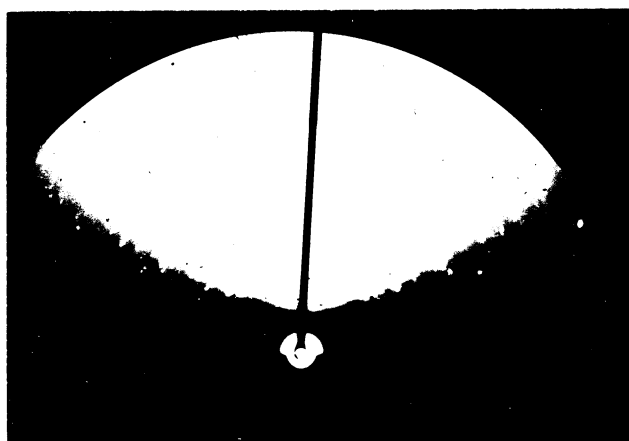


Fig. 3. Cross-section of stem of *Phytolacca* (10x).

Figures 1, 2, 3. Cross-sections of stems of *Phytolacca* (10x).

When samples 1-F and 1-J were etched strongly in the polishing solution, diffraction patterns were often obtained which contained rings belonging to the CbC-CbN and matrix phases and a number of additional weaker rings. A typical pattern of this kind is reproduced in Fig. 1b. Although the weaker rings of this pattern are difficult to measure accurately, the data obtained strongly suggest that they are produced by a carbide phase of the $M_{23}C_6$ type. It is to be emphasized that this is not a final identification and that the rings may have been produced by a material formed on the samples during the etching process. However, they do not belong to the patterns of any of the simple oxides of the alloying elements, and similar rings have not been obtained from sample 1a-A when prepared similarly to samples 1-F and 1-J.

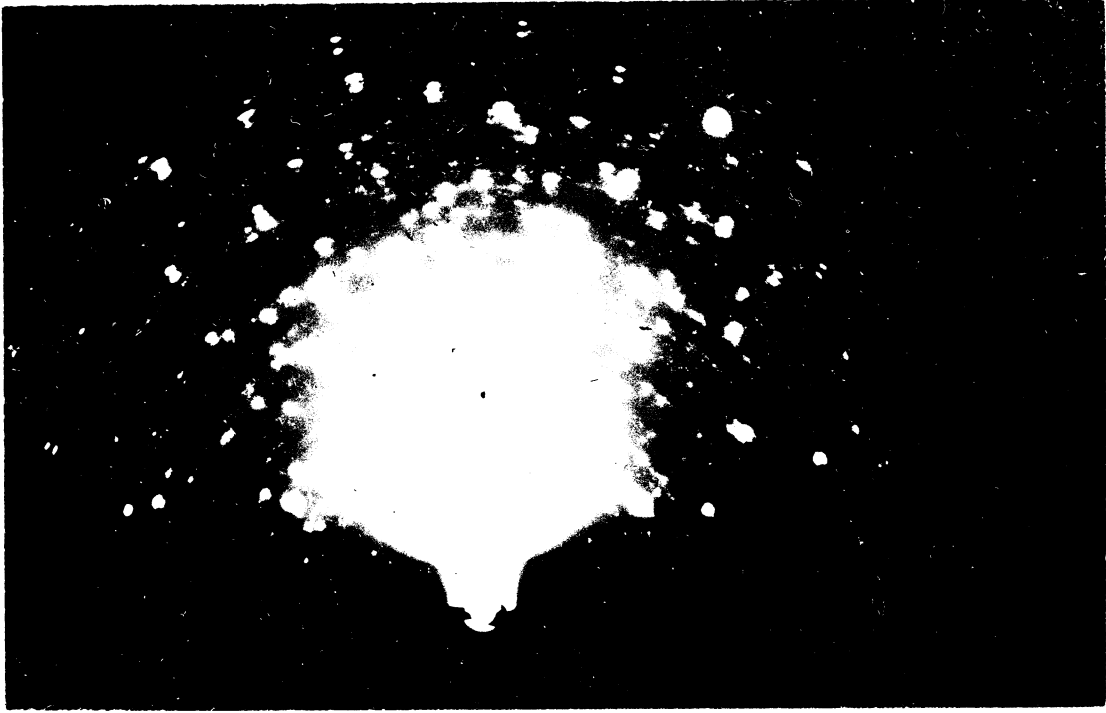
The diffraction pattern reproduced in Fig. 1c was obtained from sample 1-J, but not from the other two N-155 samples, when etched strongly in the citric acid, potassium iodide reagent. From an analysis of this pattern it appears that it was produced by a material with a crystal structure based on a face-centered cubic lattice with a unit cell edge of 6.0 Å. It has not been possible to identify this material as yet, though it is not the matrix phase of the alloy or any of the common oxides or precipitate phases reported for the elements of the alloy.

From both of the 16-25-6 alloy samples diffraction patterns were obtained containing rings which were identified as belonging to the matrix phase and to an M_6C carbide phase. No indication of other phases was found. One of these patterns is reproduced in Fig. 2.

Progress of Present Investigation

In the preliminary experiments described above, alloy samples were used which had been aged for relatively short periods. The major emphasis to date in this investigation has been placed on extending the studies to include samples of N-155 alloy, such as 1-G and 1-H, which have received long-time aging treatment, and this has proved considerably more difficult than was anticipated. The polishing electrolyte used in the preliminary experiments severely pits and etches samples 1-G and 1-H and leaves them covered with a loosely adhering, black precipitate. This precipitate is not readily soluble in the dilute acids, HCl, HNO₃, H₂SO₄, H₃PO₄, and HClO₄, nor in the rinsing agents. In an attempt to overcome this difficulty, the following polishing electrolytes have been investigated.

- (a) 70% ethyl alcohol, 20% conc. perchloric acid, 10% glycerol
- (b) 80% ethyl alcohol, 20% conc. perchloric acid



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- (c) 80% ethyl alcohol, 15% water, 5% conc. perchloric acid
- (d) 90% glacial acetic acid, 10% conc. perchloric acid
- (e) 95% glacial acetic acid, 5% conc. perchloric acid

Each of these solutions, and particularly solution (d), gives a very good polish to the samples in question without forming objectionable precipitates on them. The following solutions have been tried for use in electrolytically etching the samples after polishing with the above solutions:

- (1) 10 g chromic oxide, 90 ml water
- (2) 10 ml perchloric acid (70%), 90 ml water
- (3) 10 ml conc. nitric acid, 90 ml water
- (4) 10 g oxalic acid, 90 ml water
- (5) 45 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 100 ml water
- (6) 33 ml conc. hydrochloric acid, 67 ml ethyl alcohol
- (7) 45 g citric acid, 30 g potassium iodide, 6 ml conc. hydrochloric acid, 100 ml water.

The first five of these etching electrolytes proved unsuccessful because of a dark precipitate which formed on the alloy samples. The last two do not produce such a precipitate, but it has not yet been possible to obtain patterns of minor-phase materials by etching the samples in these solutions.

Arrangements have been completed for obtaining the use of the electron microscope facilities of the Physics Department of the University, and the preliminary work necessary to become familiar with preparing samples and replicas for electron microscopy has been completed. These facilities will be of considerable help in choosing polishing and etching procedures for use in this work, as well as in interpreting the electron diffraction patterns in terms of the microstructure of the alloys.

Work has been initiated to extend the studies on 16-25-6 alloy. When samples of this alloy are electrolyzed in solution (a), above, at current densities slightly below the optimum values for electrolytic polishing, a combined polishing and etching action is obtained. Electron diffraction patterns similar in appearance to Fig. 2 have been obtained from each of the 16-25-6 samples prepared in this way. An analysis of these patterns has not been completed.

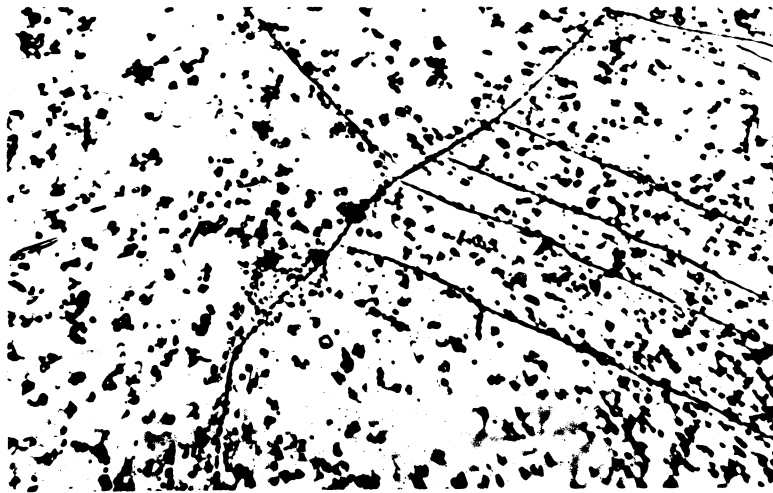
The favorable surface preparation which is obtained by the above method can be seen from Fig. 3b, which is an electron micrograph of the surface of sample 2-D. The grain boundaries and precipitate particles are clearly shown in considerable detail in this micrograph. The black object about $4 \mu\text{m}$ in diameter near one of the grain boundaries is a particle of polystyrene latex. A number of such particles are placed on the stripped collodion replica just before shadow-casting with uranium. They have uniform diameters of $2580 \text{ \AA} \pm 30 \text{ \AA}$ and aid in determining the magnification of the micrographs and the direction from which the replica was shadow-cast. The shadow of this polystyrene particle appears lighter than the surroundings and can be seen extending from the particle on the side away from the grain boundary. The shadows (light areas) of the features in the micrograph which correspond to the minor-phase particles are on the side toward the uranium source. This shows that these features are depressions in the replica and therefore that the actual minor-phase particles were protruding from the surface from which the replica was stripped. From the known diameter of the polystyrene particle, the sizes of the minor-phase particles can also be determined. Fig. 3a is an optical micrograph of the surface from which 3b was prepared.

A few preliminary experiments have been made with samples of S816 alloy, and patterns of CbC or CbN have been obtained in some cases.

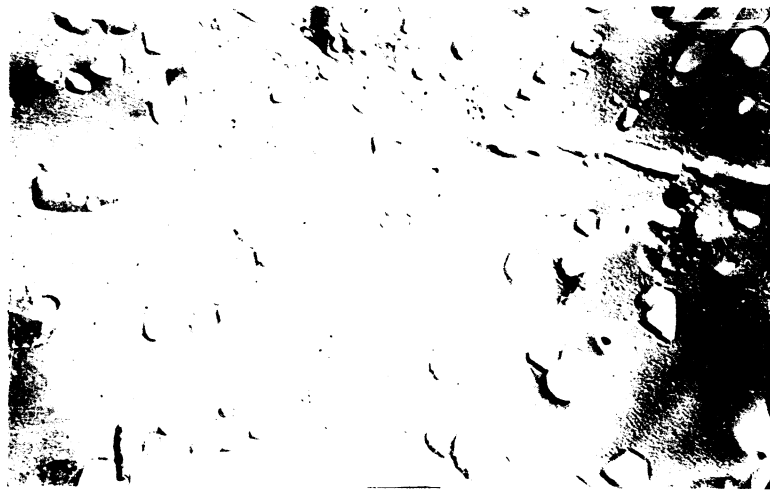
DISCUSSION

The results which have been obtained indicate that electron diffraction methods can be used successfully to detect and identify minor phases in heat-resistant alloys. A minor phase which is either CbC or CbN has been identified in S816 and low-carbon N-155 alloys and an M_6C -type carbide has been identified as a minor phase present in 16-25-6 alloy. These include all the minor phases which have been identified in these alloys by x-ray diffraction methods⁵. Strong indications of two additional phases in the N-155 alloy have been obtained by electron diffraction; namely, an $M_{26}C_3$ carbide and a face-centered cubic phase with a unit cell edge of 6.0 \AA .

The identifications have been made under somewhat more favorable circumstances than were achieved in the x-ray studies, for alloy samples were used whose previous histories are known, thus permitting a correlation of the results with those of various metallurgical studies. In this connection it has been found that the minor phase which is present in the unaged, solution-treated samples of N-155 is either CbC or CbN, and that this phase persists when the alloy is aged for periods up to 24 hours at 1600°F . Evidence has been obtained which strongly suggests that other minor phases begin to form at this point. No electron diffraction results have been obtained so far



a. Optical Micrograph, 2000X



b. Electron Micrograph, 14000X

Figure 3. Micrographs of Polished and Etched Surface of 16-25-6 Sample 2-D.

which indicate the presence of σ phase in these alloys, however. A further advantage of the electron diffraction method lies in the fact that it is not necessary to digest or destroy the alloy samples in making the identifications of the minor phases. This permits an examination of the surfaces from which the diffraction patterns are obtained by electron or optical microscopy to correlate the diffraction results with the general microstructure of the alloys. Electron microscopy is particularly useful for this purpose because of the high magnification and resolving power which it affords. It also provides direct evidence concerning the relief of the particles on the surface and may be extremely useful in determining when various minor phases are properly revealed for obtaining diffraction patterns.

In continuing this investigation it is proposed to concentrate on extending the studies of 16-25-6 and low-carbon N-155 alloys to include samples which have been aged for long periods. It appears that considerable difficulty may be encountered in finding satisfactory procedures for polishing and etching these samples. Particular emphasis will be placed on clarifying the identification of $M_{23}C_6$ as a minor phase of N-155 alloy and also in looking for evidence of the presence of σ phase in samples of this and the 16-25-6 alloy which have been mechanically worked at high temperatures. Electron micrographs will be obtained from each new sample studied and also from those which have already been investigated. The occurrence of distinct spots in the diffraction patterns suggests that they may be used in connection with the micrographs to obtain information concerning the crystallographic orientation of the precipitate particles relative to the crystals of the matrix phases of the alloys.

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