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ELECTRON METALLOGRAPHIC INVESTIGATIONS
OF HEAT-RESISTANT ALLOYS

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ABSTRACT

The work described in this report includes: (1) the development of techniques for isolating grain-boundary carbides from nickel-base alloys, using extraction replica techniques applied to fractured surfaces, to facilitate studies of the carbides by electron microscopy and identification of them by electron diffraction; (2) preliminary testing of a vacuum cathodic etching apparatus; (3) continued work to identify the minor phases of Udimet-500 alloy and to determine the role of the elements boron and zirconium on the structure and distribution of these phase in the alloy; and (4) the melting, rolling, and heat-treatment of a series of alloys of special composition preparatory to undertaking studies of the influence of cobalt and molybdenum on the structure and properties of the Udimet-500 alloy.

OBJECTIVES

Various recent metallurgical investigations indicate that the minor phases which precipitate in heat-resistant alloys during exposure to stresses and high temperatures have pronounced effects on the metallurgical properties of the alloys. To understand and control the alloys' properties better, it is therefore desirable to have detailed information on their minor phases. The work under Contract No. AF 33(616)-3250 has been undertaken to employ the highly sensitive techniques of electron diffraction and electron microscopy in 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 in 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 on 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 and microstructures.

ACKNOWLEDGMENTS

INTRODUCTION

A summary progress report was submitted in August giving a general description of the overall program of research which has geen carried out under Contract No. AF 33(616)-3250 to that date. As was indicated, the research work has been divided into two general phases along the lines outlined above. The first of these, involving work to develop improved techniques for adapting electron metallographic methods to heat-resistant alloys, has included studies of methods for preparing extraction replicas for identification of carbides in the alloys, the construction and preliminary testing of a vacuum cathodic etching apparatus, and work in developing an etchant which selectively attacks columbium and titanium carbide and nitride phases. The second phase of the work, involving the application of the electron metallographic techniques to the studies of particular alloys, included studies of the effects of vacuum- versus air-melting on microstructures of the nickel-base alloys, investigations of the mechanism by which boron and zirconium influence the properties and structures of nickel-base alloys, studies of the influence of cobalt and molybdenum on the microstructures of nickel-base alloys, correlations of hardness of commercial nickel-base alloys with their microstructures to determine the basic hardening mechanisms, and studies to identify the phases precipitating during high-temperature aging in a cobalt-base alloy designated as S816.

The work which has been carried out during the period covered by this present report has been concerned principally with four of these problems: (1) improvement of the techniques for isolating the grain-boundary carbide phases of the Udimet-500 alloy by means of extraction replicas for identification of the carbides by selected area electron diffraction techniques; (2) the investigation of the technique of vacuum cathodic etching to determine its suitability for use in connection with the electron diffraction and electron microscopic studies of the heat-resistant alloys; (3) studies of the effects of cobalt and molybdenum on the structure and properties of the nickel-base alloys; and (4) investigation of the microstructural differences between nickel-base alloys with and without boron and zirconium.

RESEARCH WORK AND RESULTS

EXTRACTION REPLICA TECHNIQUES FOR ISOLATION OF GRAIN-BOUNDARY CARBIDES

The extraction replica technique which was developed by Fisher[1] several years ago provides one of the most conclusive methods presently available for identifying the precipitate phases in alloy systems. In this method as it is conventionally used, the polished surface of the alloy specimen under investiga-

tion is given an initial etching treatment with a reagent which leaves the precipitate particles protruding from the matrix phase in the alloy, a replica film of the type used in electron microscopy is applied to the etched surface, and the surface is etched a second time, with the replica film in place. The second etching treatment serves to free the replica film and some of the smaller precipitate particles from the surface. Since the latter were protruding slightly from the surface at the time the replica film was applied, they will be partially imbedded in the film and will remain attached to it. When the films are examined in the electron microscope, the particles can be observed directly, and it is possible to obtain electron diffraction patterns from known groups of them by means of the selected area techniques of electron diffraction.

One of the principal disadvantages of this technique is that it is not well adapted for use in the identification of large precipitate particles, for these generally extend to such a depth below the surface of the sample that it is not possible to etch heavily enough to free them without damaging the replica film. This is particularly true in the case of carbides precipiated at grain boundaries, where the particles frequently join in complex networks which extend for considerable distances along the boundaries.

The results of recent metallurgical studies indicate that the high-temperature rupture strengths of the nickel-base alloys are related to the characteristics of the grain-boundary phases which precipiate during aging and testing. As a consequence, it has become highly desirable to develop methods for identifying these phases, and for studying their structures. Both reflection electron diffraction data from particles protruding from etched surfaces and x-ray diffraction data from carbide residues separated from alloys can contribute to this end. However, both of these methods suffer from the disadvantage that it is not possible to associate the diffraction patterns with particular types of particles which can be observed in the alloys by electron microscopy.

A modification of the extraction replica technique as described above has recently been devised by Plateau, Henry, and Crussard,[2] making it possible to isolate the grain-boundary precipitates with extraction replicas. This technique is based on the fact that fractures frequently follow grain goundaries, so that large numbers of grain-boundary precipiate particles are exposed in fractured surfaces. If a replica film is applied to such a surface and the surface is then etched, many of these particles remain attached to the replica as it is freed from the surface, and can then be examined by electron microscopy and electron diffraction as described above.

Figure 1 shows typical results obtained from the fractured surface of a 1/4-inch-diameter rupture test specimen of Udimet-500 alloy which was subjected to 20,000-psi stress at 1600°F for 165 hours. This treatment produced 1.2 percent elongation, but did not cause rupture. To obtain the necessary fracture surface, the specimen was cooled to liquid nitrogen temperature and broken with a hammer and chisel. The surface thus obtained was etched lightly by immersion in a mixture of 1 part of red fuming nitric acid and 3 parts hydrofluoric acid

(40%) to delineate the carbide particles. A carbon replica film was then evaporated onto the surface[3] and this was backed by a collodion film to give added strength for subsequent handling. The surface was again etched by electrolysis in a solution of 1 part phosphoric acid (85%) to four parts by volume of water until the film was freed. The composite film was then picked up from the surface of the etching bath on a microscope support grid, washed thoroughly with distilled water to remove etching reagent, and then with amyl acetate, using the method of Jaffe,[4] to remove the collodion backing layer. Finally, a thin layer of aluminum was evaporated onto the replica to provide an internal standard for subsequent electron diffraction studies.
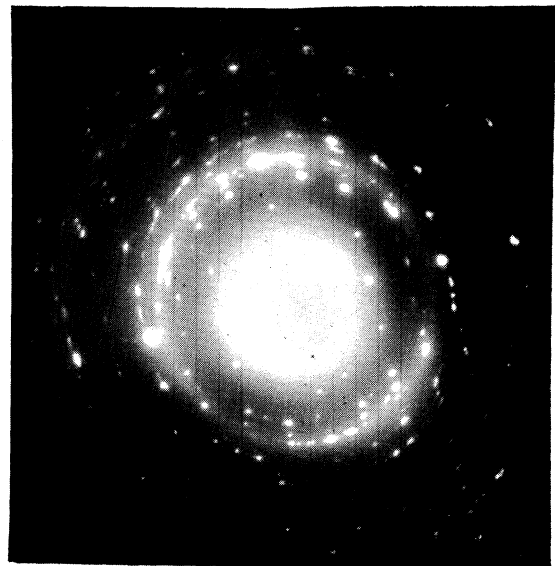
| a. Electron Micrograph. | b. Electron Diffraction Pattern. |

Figure 1. Electron micrograph (X8000) and selected area electron diffraction pattern of carbides isolated from a fractured surface of a Udimet-500 alloy by extraction replicas.

The electron micrograph of Figure 1a shows the carbide particles which were extracted in this way. It will be noted that these particles are relatively large, and that they appear to have grown together into a rather extensive network. It is therefore understandable that they are quite difficult to isolate from the alloy by the conventional extraction replica methods. The electron diffraction pattern in Figure 1b was obtained from the particles shown in the micrograph. The continuous rings are due to the aluminum standard, while the array of spots was produced by the carbide particles. Analysis of this pattern has indicated that the particles are an $M_{23}C_6$-type carbide.

This method is being used extensively in the studies of the Udimet-500 alloy, and is proving of great utility in identifying the grain-boundary precipitates which are believed to be important in controlling the rupture strength

of this alloy. It is believed that this method can be useful in many cases as a supplement to other methods, to provide more discrimination in determining the location and distribution of precipitated phases in alloys. The only major difficulty which has been encountered arises from the fact that in many cases the extracted carbide particles are too large to transmit the electron beam, and then electron diffraction patterns cannot be obtained from them. A possible solution to this problem would be to treat the particles with reagents which would reduce their size sufficiently to permit penetration by the electron beam. Alternately some increase in penetration can be achieved by increasing the accelerating potential used in generating the electron beam. The instrument which has been used in this work operates at a maximum accelerating potential of 50 Kv and therefore has less penetrating power than other commercially available instruments which operate at 80 or 100 Kv.

VACUUM CATHODIC ETCHING

Vacuum cathodic etching consists of removal of material from the surface being etched by bombardment with gas ions, usually of one of the inert gases or nitrogen. The particular advantages of the method compared to chemical or electrolytic methods are: it can be adapted to a wide variety of different materials with only minor variations in etching conditions; because the etching is done in an inert atmosphere at reduced pressure, relatively reactive metals can be etched without oxidation or corrosion; it is readily applicable to porous materials; combinations of materials of different activities, as in plated or fused couples and mixtures such as ceremets, can be etched as easily as more homogeneous systems; and etched surfaces are virtually never contaminated with residues, deposits, and corrosion products as is frequently the case in conventional etching methods. Although this method has been known for some time, it has not been widely used in the past because of the complicated and expensive apparatus that was required, and the rather erratic results that were frequently obtained. Recently, however, interest in it has been revived, because apparatus of simplified design has been developed,[5,6] and a better understanding of the variables controlling the etching has been achieved.

Vacuum cathodic etching is of particular interest as a method of etching the heat resistant alloys for electron metallographic examinations, since it could provide a means of overcoming the problems of surface contamination which are so consistently encountered when attempting to etch these alloys by conventional methods. Therefore a vacuum cathodic etching apparatus has been constructed and work is in progress to determine whether the characteristics of the surfaces produced are such that satisfactory electron diffraction patterns and electron micrographs can be obtained from them.

The apparatus is based on a simplified design which was development by J. B. Newkirk and W. G. Martin.[6] Its essential features are shown diagrammatically in Figure 2. The etching chamber is a 3-liter pyrex flask with large ground-glass joints for inserting the anode and the cathode specimen support. The
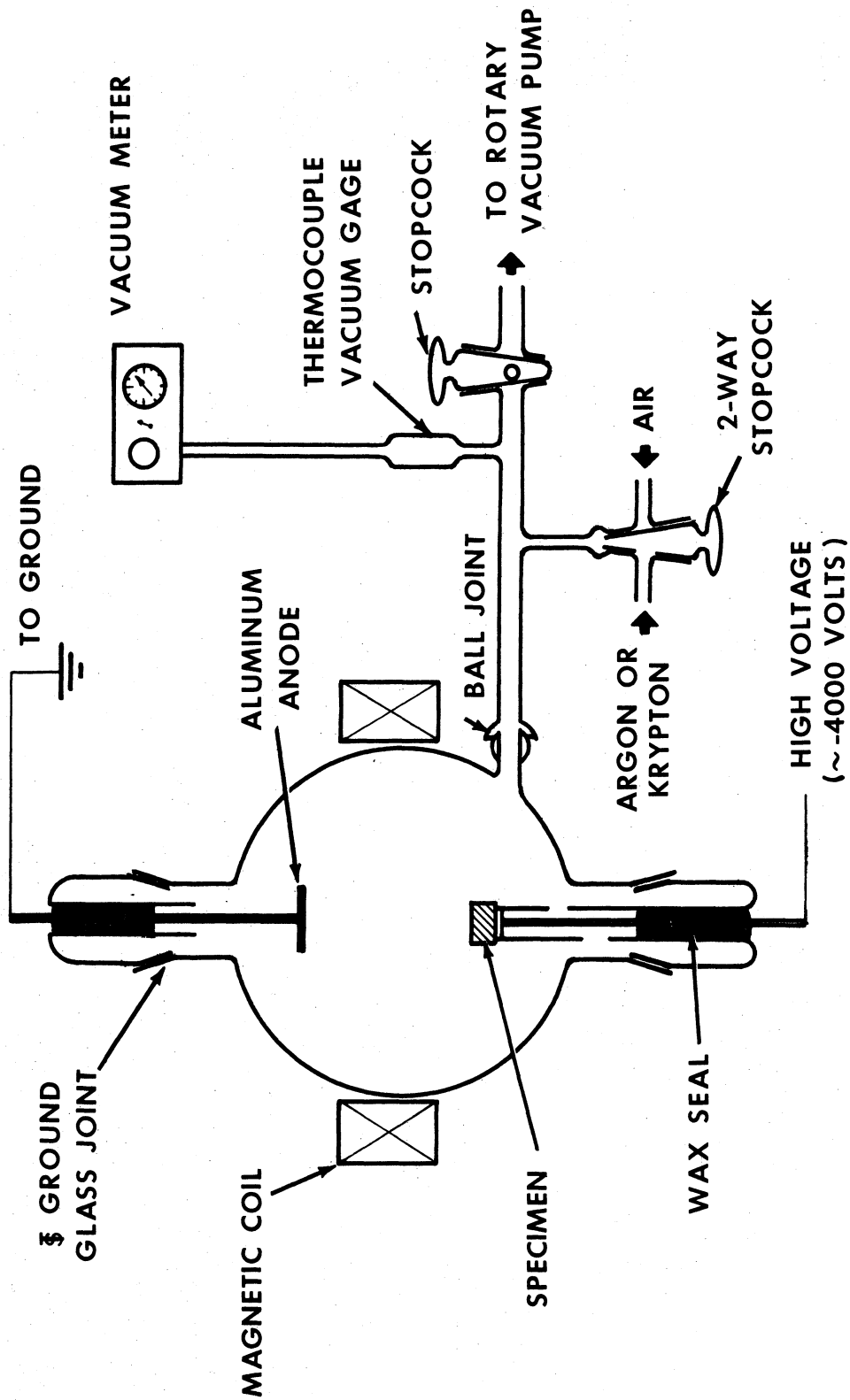
Figure 2. Schematic diagram of vacuum cathodic etching apparatus.

chamber is evacuated by a rotary oil pump through a manifold equipped with the necessary stopcocks for admitting the etching gas. Gas pressure in the chamber is measured by a thermocouple vacuum gauge. The potential for generating the discharge is supplied by the filtered power supply whose circuit is given in Figure 3. This supply is capable of delivering up to 20 milliamperes of current with a maximum potential of about 6500 volts. An added feature of the apparatus is the magnetic coil around the etching chamber which serves to increase the path of the electrons, thus increasing their efficiency in producing ions. Newkirk and Martin[6] claim that this greatly reduces the time required for etching at a given pressure of gas in the chamber.

In operation, the specimen to be etched is placed on the cathode, and the assembly is inserted into the etching chamber. Air is removed from the chamber by alternately evacuating it to a few microns pressure and introducing argon gas, two or three times. Then the chamber is evacuated, the etching gas is introduced to a pressure nearly equal to atmospheric pressure (Newkirk and Martin recommend krypton, although argon has been used by most other workers), and the chamber is then evacuated to the pressure desired for the etching operation (usually in the range of 10 to 50 microns). The chamber is then sealed off by closing the stopcock in the pumping line, the magnetic coil is turned on, and the high potential is turned up until the desired ion current is obtained. Usually a current in the range of 2 to 10 microamperes and a potential in the range 4000 to 6000 volts are used. At a given voltage, the ion current obtained when a steady discharge is established will depend on the gas pressure in the chamber. During the course of the etching, the pressure frequently rises due to heating effects and gas evolution from the specimen, and it is then necessary to open the stopcock for a short period and reduce the pressure to the desired value.

The work to date on this etching method has been concerned largely with constructing and testing the apparatus. Several unusual difficulties have been encountered which delayed this phase of the program more than was expected. When the vacuum system was first assembled it was not possible to maintain a satisfactory static vacuum. This was finally found to be associated with a metal thermocouple gauge which was attached to the glass manifold through a metal-to-glass seal: either the assembly leaked or out-gassed sufficiently to cause a slow but steady increase in pressure in the system, which was sufficient to be undesirable for etching. This was overcome by using an RCA type 1946 thermocouple gauge tube which has a glass envelope and can be sealed directly to the manifold. When the power supply was first tested, intense momentary discharges occurred between the anode and cathode at a voltage considerably below that required to produce the sustained discharge required for etching. This was finally traced to surge currents in the power supply, and was cured by adding the surge-limiting choke to the filter circuit. Normally, during etching the glow discharge is confined to the region between the anode and cathode in the etching chamber. Recently, however, the discharge has been observed to extend over a distance of several feet through the manifold to the grounded elements of the thermocouple tube whenever etching currents above a minimal value are

Figure 3. Circuit diagram of high-voltage power supply for vacuum cathodic etching apparatus.
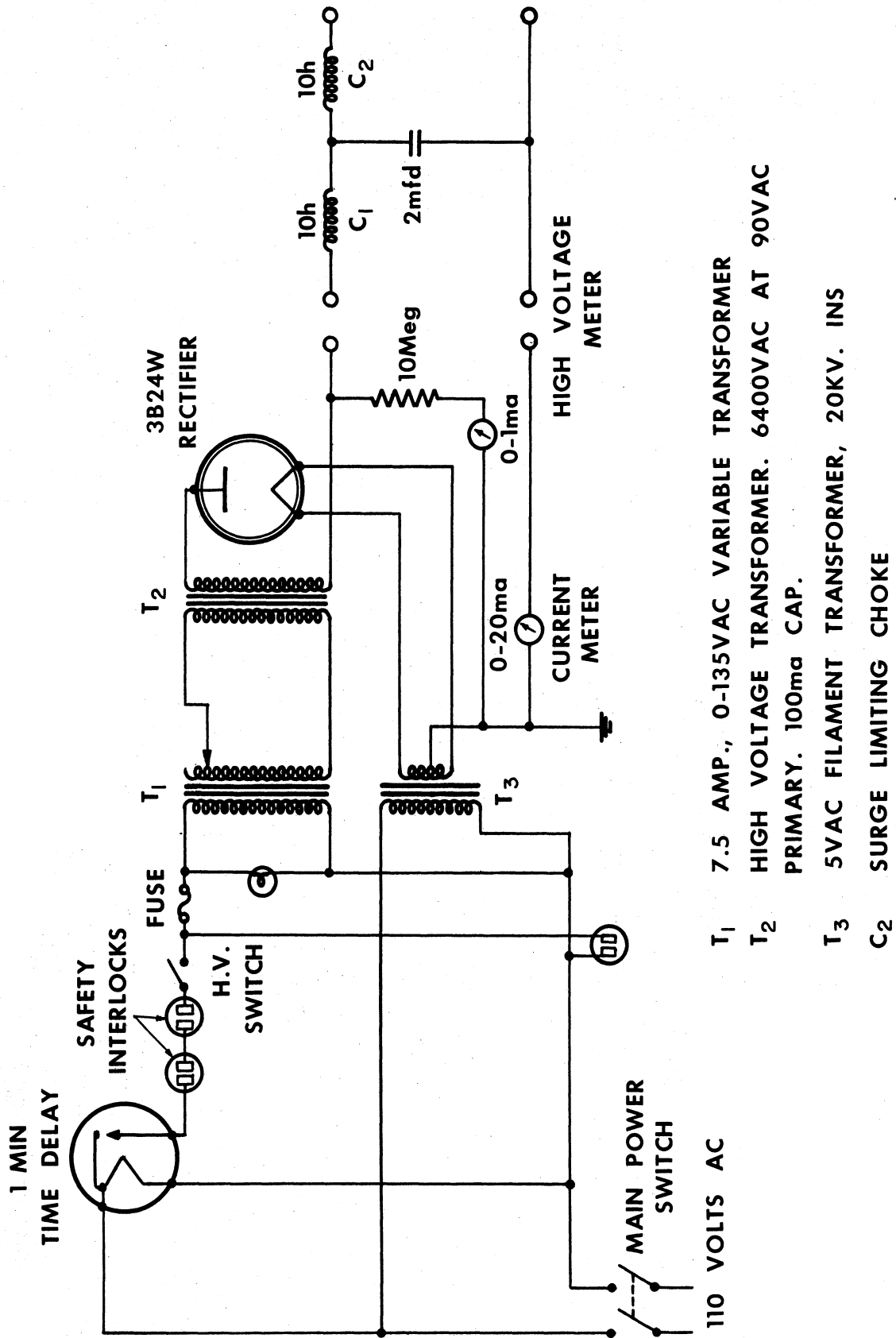
T_1 7.5 AMP., 0-135VAC VARIABLE TRANSFORMER

T_2 HIGH VOLTAGE TRANSFORMER. 6400VAC AT 90VAC PRIMARY. 100ma CAP.

T_3 5VAC FILAMENT TRANSFORMER, 20KV. INS

C_2 SURGE LIMITING CHOKE

used. The exact cause of this is not known, although it is thought to be caused by contamination of the etching chamber with a slightly volatile, easily ionized material, possibly oil from the vacuum pump or material released from the speci-ments by the ion bombardment.

In the course of the work to date it has been concluded that certain modi-fications in the apparatus would improve its performance and convenience of op-eration. Perhaps the most important of these modifications involves the intro-duction of a glass shield around the cathode assembly and specimen. Prelimin-ary experiments indicate that such a shield, if allowed to extend about an inch above the specimen, acts to concentrate the discharge on the upper surface of the specimen, and thus greatly increases the etching efficiency. It also col-lects most of the metal which is discharged from the specimen, reducing the gen-eral contamination of the etching chamber. Secondly, it seems desirable to re-duce the diameter of the etching chamber. This would permit a coil of smaller diameter to be used, thus concentrating the magnetic field more in the region between the electrodes and increasing its efficiency in enhancing ionization of the gas. It would also markedly reduce the time required to evacuate the appa-ratus. Thirdly, it is believed that more satisfactory control of the etching atmosphere could be achieved by continuously pumping on the system and regula-ting the pressure by introducing the etching gas through a controlled leak. Fourthly, it seems desirable to provide a means of cooling the specimen and the cathode assembly to reduce the possibility of gas evolution from the wax seals and structural damage to the specimen from the heat generated during the etching.

Even without these modifications, etching results highly satisfactory for optical microscopic studies of 16-25-6 and S816 alloys have been obtained. Work is now being undertaken to compare vacuum cathodically etched surfaces of these alloys with surfaces etched by usual chemical means, to determine the relative advantages of the two etching methods for electron metallographic purposes.

EFFECTS OF BORON AND ZIRCONIUM ON ALLOY PROPERTIES

In recent studies of the variability of the high-temperature strengths of nickel-base alloys, Decker and Freeman[7,8] showed that the addition of small amounts of the elements boron and zirconium greatly increases the creep-rupture strength of these alloys. In their work, a 55Ni-20Cr-15Co-4Mo-3Ti-3Al alloy was found to have a rupture life of only about 50 hours at 1600°F and 25,000 psi when free of these elements, while the addition of 0.1 percent by weight of zir-conium increased the rupture life to 150 hours, and addition of 0.01 percent of boron increased it to 400 hours. When 0.1 percent of zirconium was added to-gether with 0.01 percent boron the rupture life increased still further to nearly 600 hours.

Optical and electron microscopic examinations were made to determined the microstructural basis for these effects, and it was concluded that they were

related primarily to structural differences which developed in the grain boundaries during aging under stress. Examination of specimens aged without stress revealed no significant difference in the microstructure of the alloy, whether boron and zirconium were present or absent. When aged under stress, as in the stress-rupture tests, however, specimens without boron and zirconium characteristically developed areas along the grain boundaries which were devoid of the γ' precipitate particles, and numerous microcracks occurred between the depleted matrix and the grain-boundary carbides in these areas. In specimens containing boron and zirconium, the development of the depleted areas and microcracks along the grain boundaries was greatly retarded, and the grain-boundary carbides appeared to be enveloped in a more or less continuous film of γ' phase. In addition, unusual aggregates of angular particles, about 1000 $\overset{\circ}{A}$ in length, developed within the grains of these specimens. From their appearance these were deduced to be aggregates of carbide particles, probably of the $M_{23}C_6$ type, though possibly involving boron or zirconium in some manner.

These studies by Decker and Freeman have made the important contribution of establishing the role of boron and zirconium in controlling the high-temperature properties of the nickel-base alloys, and the overall microstructural changes through which the control is operative. In addition, they raised the very interesting problem of determining the mechanism by which such minute quantities of these elements can effect such extensive changes in the microstructures and properties of the alloys. With this problem in mind, work has been undertaken on this project to identify the minor phases occurring in the alloy used by Decker and Freeman, and to look for differences between the phases which develop when boron and zirconium are present and absent. In this work, electron diffraction examinations of etched surfaces and extraction replicas are being used, in addition to x-ray diffraction studies of residues separated from the alloys by chemically dissolving the matrix metal.

To date x-ray studies have been made of specimens aged 500 hours at 1600°F from the heats containing no boron or zirconium, 0.1 percent zirconium, 0.01 percent boron, and 0.1 percent zirconium plus 0.01 percent boron. Unfortunately, it has not been possible to make satisfactory identification of the phases in these specimens because of the complexity of the x-ray diffraction patterns obtained. In all cases the patterns contain fairly strong diffraction lines which correspond to the reflections of the TiC phase, medium strong lines corresponding to those of $M_{23}C_6$, medium weak lines corresponding to M_6C, and several weak lines corresponding to TiN. It is also possible that Cr_7C_3 is present, since the strong lines of the pattern of this phase coincide with some of those of the four phases just listed. In addition three or four very weak lines, two of which frequently have d values close to 2.22 and 2.02 $\overset{\circ}{A}$, are present in all the patterns. To date it has not been possible to assign these lines to any of the phases which are considered likely to occur in the alloy. Attempts are being made to overcome these difficulties. One approach that seems promising is to study specimens aged for shorter and longer periods at 1600°F, and also at different temperatures, in the hope that one or more of the phases will be absent or present in much larger amounts. This would permit definite assignment of

some of the lines, and aid in assigning the others. It is also possible that some fractionation of the extracted residues can be achieved by flotation or similar physical techniques.

Selected area electron diffraction examinations have been made of carbide particles separated by extraction replicas from the fractured surfaces of a specimen containing 0.01 percent boron and no zirconium which was stressed 182 hours at 28,000 psi and 1600°F, and also from a specimen containing no boron or zirconium which was stressed 165 hours at 20,000 psi and 1600°F. In each case the stress treatment resulted in 1.2 percent elongation of the specimen. Here too, some difficulties have been encountered due to the fact that most of the carbide particles are much too large to transmit the electron beam, and there-fore diffraction patterns cannot be obtained from them. This was particularly true of carbides from the specimen containing boron and zirconium. By careful examination of the replica a few groups of particles were found which were small enough to produce patterns, and these were identified as $M_{23}C_6$ particles, with some indications of M_6C. A typical micrograph showing these particles is re-produced in Figure 4. (X-ray patterns from residues from this specimen con-tained weak lines of TiC, very weak lines of $M_{23}C_6$, and medium lines of M_6C.) The carbide particles from the specimen containing no boron or zirconium were generally thinner, and gave good diffraction patterns (Figure 1). From these, $M_{23}C_6$ has been identified but no indications have been obtained of the presence of M_6C.
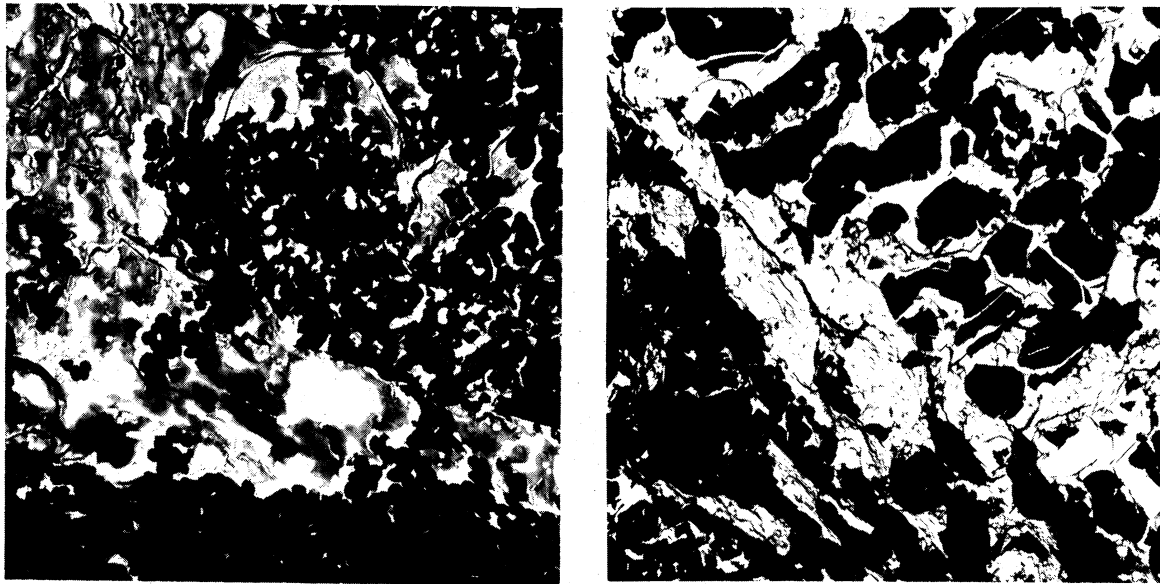

Figure 4. Electron micrographs (X8000) of carbide particles isolated from frac-tured surfaces of a 55Ni-20Cr-15Co-4Mo-3Ti-3Al alloy by means of extraction re-plicas.

Attempts have also been made to identify the aggregates of angular parti-cles which were observed in the specimens containing boron and zirconium after stress-aging. By using extraordinarily heavy etching treatments it has been

possible to isolate some of these aggregates in extraction replicas. Again, it has not been possible to obtain electron diffraction patterns because the particles are too thick to transmit the electron beam.

This inability to obtain diffraction patterns appears to be the major obstacle preventing a detailed identification of the minor phases of the boron alloys, for adequate methods have now been developed for isolating the phases from the matrix for the diffraction studies. As noted previously, this difficulty could be overcome either by use of chemical reagents to reduce the size of the particles, or by using an instrument which operates at a higher accelerating potential and which would therefore have greater penetrating power. Both of these possibilities are being actively explored.

EFFECTS OF Mo AND Co ON ALLOY STRUCTURES

Heretofore, the principal interest in studying the nickel-base alloys has been in attempting to determine the cause of apparently random variability in properties of heats prepared and processed under nearly identical conditions. In this work much attention has been given to the role of titanium and aluminum, because of the precipitation-hardening effect of the γ' phase which they produce, and to other elements such as oxygen, nitrogen, and carbon which are present in small quantities, because of the inherent difficulties in controlling the amounts and distribution of these elements. However, little attention has been given to the effects of the major alloying constituents such as cobalt and molybdenum. The recent results obtained by Decker and Freeman[7,8] indicate that the property variations are largely due to previously unrecognized variations in the amounts of boron and zirconium in the alloys, and that when these elements are controlled, the properties of a given alloy become satisfactorily reproducible. This now opens the possibility of making meaningful studies of the effects of major alloying constituents such as Fe, Co, and Mo which are present in different amounts in various alloys.

Some work had been previously started along these lines, using a series of special alloys designated 1093, 1094, and 1128. This work was summarized in the previous report, but was not carried beyond the stages of preliminary investigations of microstructural differences, because it was realized the alloys had been prepared prior to the time that the role of boron and zirconium was recognized, and that no precautions were taken to control these elements. Consequently, it was decided to prepare new alloys under more carefully controlled conditions, and during the past three months, the melting, rolling, and heat-treating of these have been accomplished.

The aim compositions of the alloys are given in Table I, together with the aim composition of Udimet-500 alloy. It will be noted that the experimental alloys are based on the Udimet composition, but differ from it in the amounts of Co and Mo. It is believed that studies of this series of alloys will provide an insight into the role which these elements play in determining the properties of the Udimet-500 alloy.

TABLE I. AIM COMPOSITIONS OF SPECIAL ALLOYS
(Weight percent)

Alloy	Ni	Cr	Al	Ti	Co	Mo	B	C
1179	77.2	22.8	-	-	--	-	0.1	.005
1180	74	20	3	3	--	-	0.1	.005
1181	59	20	3	3	15	-	0.1	.005
1182	70	20	3	3	--	4	0.1	.005
Udimet-500	55	20	3	3	15	4	0.1	.005

The experimental alloys were melted in a vacuum furnace at an average pressure below 1 micron of Hg. An MgO crucible was used to eliminate zirconium pick-up during melting, and none of this element was added to the alloy. Virgin melting stock consisting of electrolytic nickel, electrolytic chromium, electrolytic cobalt, arc-melted molybdenum, Ti55A-grade titanium, 99.99 percent aluminum pig, and spectrographically pure carbon was used. The boron was added as NiB. In the procedure generally followed, the crucible was charged with the nickel, chromium, cobalt, and carbon, and the molybdenum, aluminum, titanium, and NiB were added a few minutes prior to pouring, after the charge was melted and out-gassed. The 10-pound ingots thus obtained were homogenized 2 hours at 2150°F, surface ground, and then rolled to 7/8-inch-square bar stock from 2150°F, in about 25 passes, each producing about 7-percent reduction in area, with 10-minute reheats at 2150°F between passes. The 7/8-inch billet was then cut in thirds, and each part further reduced to 1/2-inch-square bar stock in a similar manner.

The program of study proposed for these alloys, including the Udimet-500, is as follows:

1. Investigation of variations in solution temperature using specimens treated 4 hours at 1800°F, 4 hours at 1975°F, and 2 hours at 2150°F.

2. Investigation of effects of aging on microstructure, minor phases, and hardness, using specimens aged 1, 4, 10, 30, 100, and 400 hours at 1200°, 1400°, and 1600°F, and 1, 4, and 10 hours at 1800°, 1900°, and 2000°F, after solution treatment at 2150°F and air cooling.

3. Investigation of effects of aging under stress. For this purpose, alloys would be tested to determine their relative strengths at a temperature in the range of 1500-1600°F and at a stress level in the neighborhood of 25,000 psi, and examinations would be made to determine the microstructures and minor phases of the ruptured specimens. In all probability it would be desirable in some cases to employ interrupted rupture tests also, to compare the rate of development of significant microstructural feature during testing.

At present specimens are being aged for the first two parts of this program, but it is not considered possible to complete any appreciable part of the third part before termination of the present contract.

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