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THE EFFECT OF HIGH-TEMPERATURE AGING ON THE DEVELOPMENT
OF MINOR PHASES IN INCONEL-X ALLOY*

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ABSTRACT

Specimens of Inconel-X alloy solution-treated at 2050°F and aged for periods of 1, 10, 100, and 1000 hours at 1200°, 1400°, and 1600°F have been examined by electron microscopy and by electron and x-ray diffraction. Four minor phases were identified: titanium nitride, niobium nitride, an $M_{23}C_6$ complex carbide, and the intermetallic γ' phase. The two nitrides were found in all of the aged specimens and in the solution treated alloy, the γ' phase formed at all aging temperatures, while the $M_{23}C_6$ carbide formed only on aging at 1200° and 1400°F. Variations in the metallurgical properties of the alloy are correlated with the size and distribution of the γ' particles.

The nickel-chromium alloys containing titanium and aluminum comprise one of the most promising groups of alloys currently available for applications requiring high strength and corrosion resistance at elevated temperatures. These alloys are of particular interest because exposure to temperatures in the range of 1200° to 1500°F brings about precipitation processes which markedly improve their strength and hardness and increase their service life. Consequently, they are widely used in the manufacture of components of gas turbines and jet aircraft engines where prolonged service in this temperature range is desired.

The present investigation was undertaken to identify the minor phases and to study the microstructural changes which occur in a typical commercial alloy of this group as a result of aging treatments at these high temperatures. Inconel-X alloy was selected for this investigation because the previous studies of Frey, Freeman, and White¹ have provided extensive information on the variations in metallurgical properties of this alloy resulting from such aging treatments, and because aged specimens of the alloy prepared by these investigators were made available for present use. The techniques of x-ray and electron diffraction were used in identifying the minor phases, and electron microscopy was used to study the microstructure.

Several recent investigations have provided a very useful background of information for this work. Taylor and Floyd^{2,3,4} have made extensive studies of the phases of alloys in the nickel-rich portions of the nickel-chromium-aluminum, nickel-chromium-titanium, and nickel-titanium-aluminum systems. In addition to the austenitic γ matrix phase, they found intermetallic γ' and η phases in the nickel-chromium-aluminum and nickel-chromium-titanium alloys, respectively. The η phase was found to have a hexagonal structure based on that of Ni_3Ti , while the γ' phase was found to have a face-centered cubic structure based on that of the Ni_3Al phase of the nickel-aluminum system. Both the η and γ' phases were observed in the nickel-titanium-aluminum alloys. The lattice parameter of the γ' phase, though always very close to that of the matrix phase, was found to vary somewhat with the titanium content, indicating that it was capable of dissolving some titanium. More recently Nordheim and Grant⁵ have identified both of these phases in quaternary alloys containing a 4:1 ratio of nickel to chromium with varying amounts of titanium and aluminum up to 4%. They also concurred with Hignett⁶ in concluding that the high strength of the titanium-aluminum-containing nickel-chromium alloys was due to the precipitation of the γ' phase. Rosenbaum⁷ has reported the identification

of titanium and niobium nitrides in hot-rolled specimens of the Inconel-X alloy, and Beattie and VerSnyder⁸ have reported the identification of titanium carbonitride, M_6C , $Cr_{23}C_6$, Cr_7C_3 , Cr_3C_2 , and M_2Ti as minor phases of M252 alloy. The metallurgical studies of Frey, Freeman, and White¹ on Inconel-X alloy provided the basis for the present investigation and will be discussed in connection with results of this investigation.

MATERIALS AND METHODS

The specimens of Inconel-X alloy prepared by Frey, Freeman, and White¹ were taken from 1-inch, round bar stock of a commercial heat having the following weight composition: 0.04% C, 0.56% Mn, 6.59% Fe, 0.007% S, 0.38% Si, 0.03% Cu, 73.22% Ni, 14.97% Cr, 0.78% Al, 2.38% Ti, and 1.02% Nb. All specimens were solution-treated at 2050°F for four hours and then water quenched. Those examined in the present investigation included one solution-treated (unaged) piece and specimens subsequently aged for 1, 10, 100, and 1000 hours at 1200°, 1400°, and 1600°F.

Electron diffraction techniques were employed in identifying the minor phases by directing the electron beam across surfaces which had been especially polished and etched so that the minor-phase particles protruded slightly above the matrix metal. Due to the low penetrating power of the electrons, diffraction patterns obtained in this manner arise almost entirely from the protruding minor-phase particles. These patterns are analogous to x-ray powder diffraction patterns and by comparison with x-ray powder data can be used to identify the minor phases. This general procedure was developed by Heidenreich, Sturkey, and Woods⁹ in studies of magnesium, aluminum, and copper alloys; its adaptation to the heat-resistant alloys has been discussed in a recent publication by the present authors¹⁰. The particular advantage of this method arises from the high sensitivity of electron diffraction which permits very small quantities of minor phases to be detected and identified without completely separating them from the matrix phase. In addition, electron or optical microscopic studies may be made of the etched surfaces used in the diffraction studies, permitting direct correlation of the minor phases identified with the microstructural characteristics of the specimens.

The Inconel-X alloy specimens were prepared for the electron diffraction studies by electrolytic polishing in an electrolyte consisting of 10 ml of perchloric acid (68 - 72%) and 90 ml of glacial acetic acid, followed by immersion etching in aqua regia or by electrolytic etching in one of the following reagents: A - 33 ml hydrochloric acid and 67 ml glycerol; B - 5 ml hydrofluoric acid (48%), 10 ml glycerol, and 85 ml ethyl alcohol (95%). For unmounted specimens about 1 x 1/2 x 1/4 in. in size, the polishing operation required about 30 - 90 seconds of electrolysis

at currents of 4 to 6 amperes, depending on the characteristics of the individual specimens. The electrolysis was usually carried out in several periods of 5 - 15 seconds each to reduce pitting of the surfaces and to avoid overheating the perchloric acid solution. The etching treatments were usually of 5 - 30 seconds duration with currents of 0.1 - 0.3 ampere. Immediately after etching, specimens were thoroughly rinsed with distilled water, then with a 50 - 50 mixture of distilled methanol and acetone, and then allowed to drain on clean filter paper and to dry by evaporation.

X-ray diffraction examinations were made both on bulk specimens of the alloy and on residues separated from the specimens by digestion of the matrix phase. The bulk specimens were examined specifically to detect the intermetallic γ' phase. These examinations were carried out on surfaces of the specimens which were electrolytically polished and etched, using the procedures described above, to minimize the possibilities of effects from surface disturbances. High-resolution, back-reflection techniques were used to obtain maximum resolution of the reflections from the γ' and γ (matrix) phases, and Cu-K α radiation was employed to reduce background and fluorescent scattering. The residues were examined to obtain diffraction data from the insoluble carbide and nitride phases which would supplement the data obtained on these phases by electron diffraction. These residues were obtained by immersing the specimens for 24 hours in a 10% solution of bromine in anhydrous methanol¹¹ to dissolve some of the matrix metal. The undissolved portions of the specimens were then removed, the bromine reagent decanted off, and the insoluble residues remaining in the containers were washed repeatedly with methanol and dried. The digestion and washing operations were carried out in centrifuge tubes, and centrifugation was used to aid in separating the residues from the liquids. The dry residues were mounted in thin glass capillaries and the diffraction patterns were taken in a Straumanis type camera of 114.6 mm diameter, using Cu-K α radiation.

The electron microscopic studies of the microstructures of the aged specimens were made on the same surfaces as were used in the electron diffraction studies. Of the three etchants used in preparing these surfaces, reagent B, above, gave the most satisfactory results for microstructural purposes. All of the micrographs reproduced in this paper to show the general microstructural characteristics of the different specimens were taken from surfaces etched with this reagent. A special etching procedure producing a preferential attack on the γ' particles was also used to determine the distribution of the γ' phase in the alloy. This procedure has been described in detail elsewhere.¹² Briefly, it consists of etching surfaces prepared with reagent B, for a very short period in a solution consisting of 5 ml of hydrofluoric acid (40%), 10 ml of glycerol, 10 - 50 ml of ethyl alcohol, and water to make 100 ml total volume. The etching is carried out electrolytically at a low current density, and must be carefully controlled to avoid an excessively heavy attack on the γ' particles.

The electron micrographs were obtained from the etched surfaces through the use of negative collodion replicas. These replicas were separated from the surfaces by the dry-stripping procedure and were shadowcast with palladium to increase their contrast. Prior to shadowcasting, polystyrene latex spheres having uniform diameters of about 2600 Å were placed on the replica surfaces to provide a size standard from which the magnifications of the micrographs can easily be determined.¹³ These spheres also aid in determining the angle and direction of the shadowing and are useful objects to observe in focusing the microscope when photographing the micrographs. For reproduction here, the micrographs were photographically processed so that the shadows formed in the shadowcasting operation appear black, as on the original photographic plates exposed in the microscope.

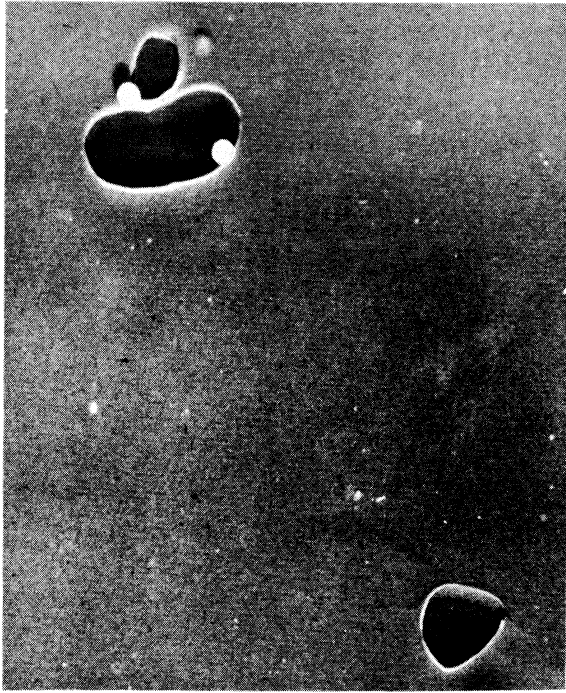
OBSERVATIONS

MICROSTRUCTURE

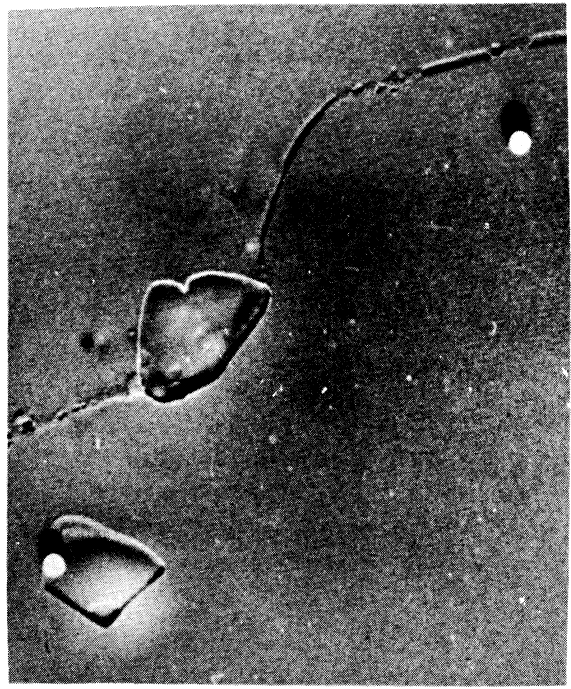
Electron microscopic examinations were made of all of the aged specimens and of a specimen which was solution-treated and water-quenched, but not aged. Typical electron micrographs are reproduced in Figs. 1-3. They represent all of the specimens except the one aged for only one hour at 1200°F, whose microstructure is very similar to that of the unaged specimen.

The micrographs from this latter specimen and from the unaged specimen showed a few widely separated precipitate particles, indicating that the solution treatment at 2050°F was not entirely effective in eliminating precipitates from the alloy. These particles were either spheroidal or showed only two or three surface planes, and in many cases exceeded 30,000 Å in diameter. They were not particularly prevalent at the grain boundaries, but rather appeared to be randomly distributed throughout the alloy. Their large size and random distribution suggest that they were stable at the 2050°F solution-treating temperature and formed either during solution treatment or during some previous high-temperature manufacturing process. Apart from these particles, however, no minor phases were observed in these specimens.

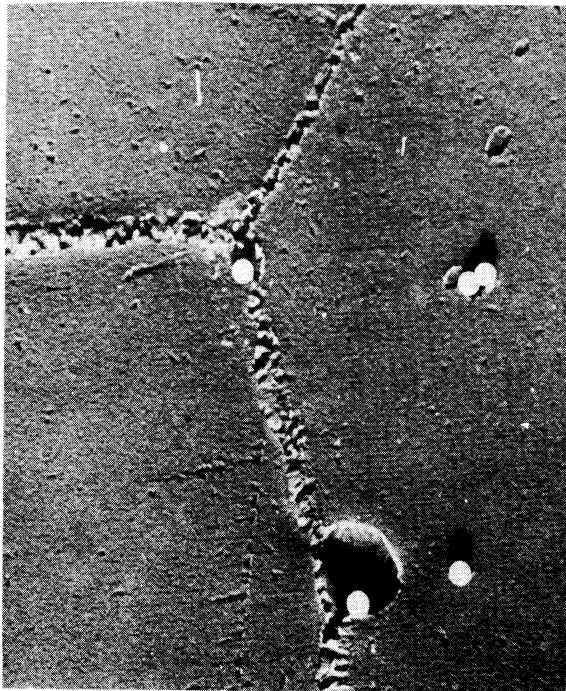
The subsequent aging treatments at 1200°, 1400°, and 1600°F appeared to have little effect on the number or distribution of the above particles, though long periods of aging, particularly at 1400° and 1600°F, appeared to cause some increase in their size. The most marked effect of the aging treatments was to cause additional precipitation both at the grain boundaries and within the matrix grains. The development of this precipitation varied considerably with the aging temperature.



a. Unaged.



b. Aged 10 hours.



c. Aged 100 hours.



d. Aged 1000 hours.

Fig. 1. Electron micrographs of Inconel-X specimens solution-treated 4 hours at 2050°F and aged at 1200°F. X 12,000.



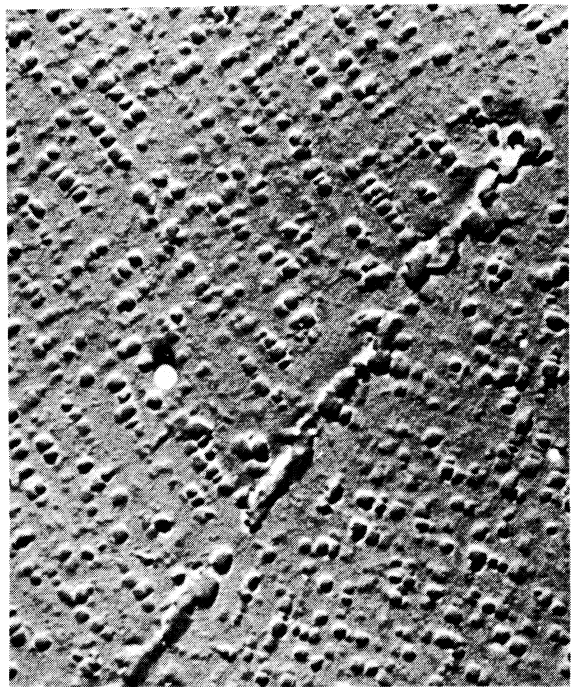
a. Aged 1 hour.



b. Aged 10 hours.

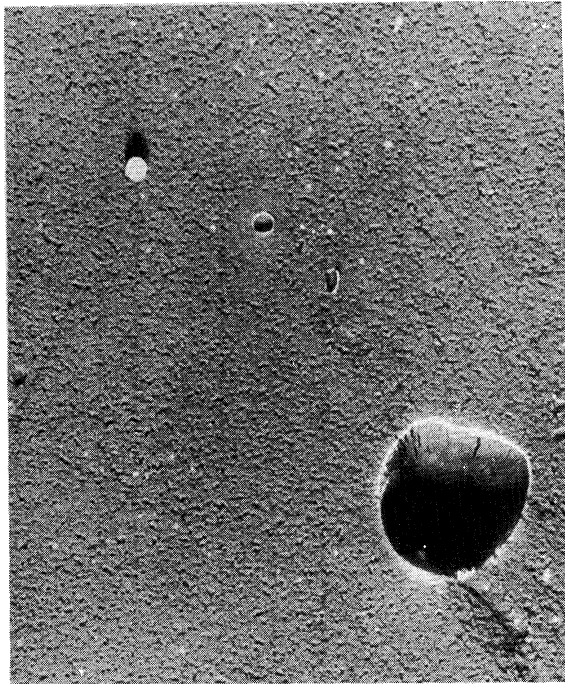


c. Aged 100 hours.

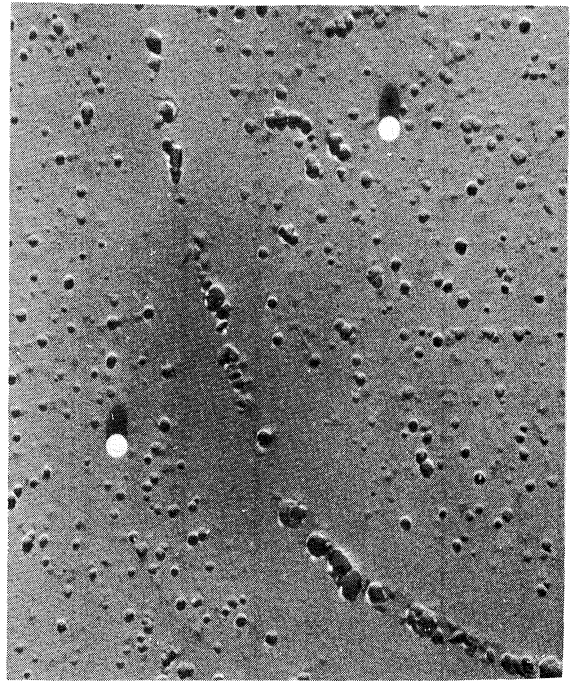


d. Aged 1000 hours.

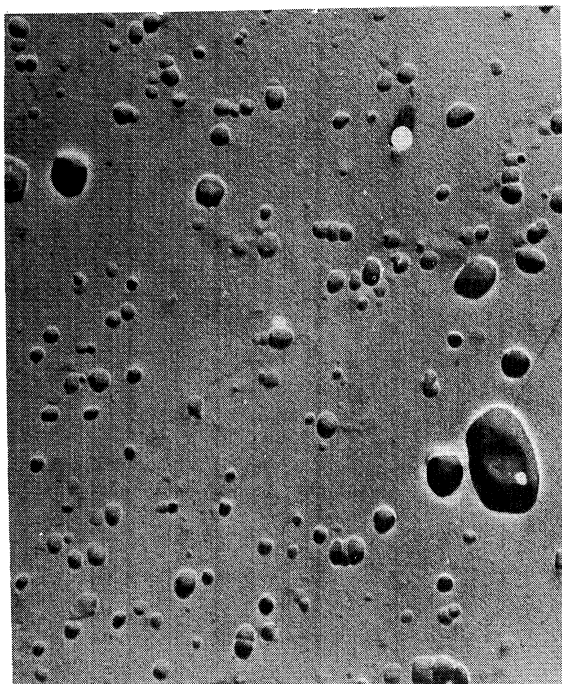
Fig. 2. Electron micrographs of Inconel-X specimens solution-treated 4 hours at 2050°F and aged at 1400°F. X 12,000.



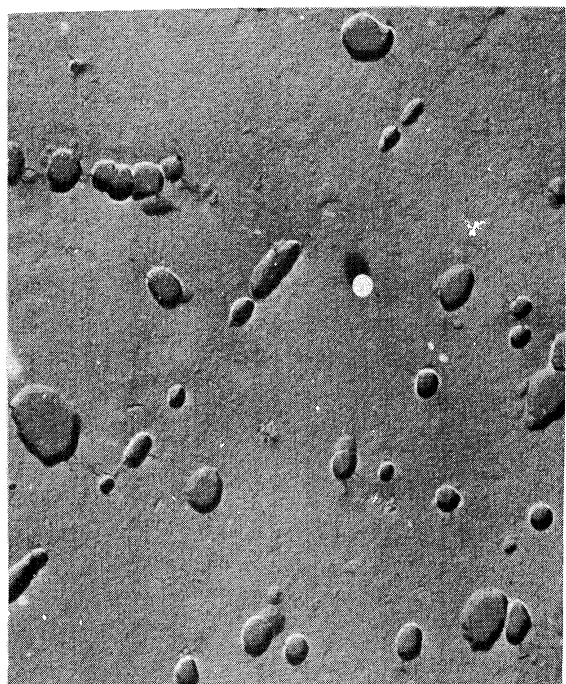
a. Aged 1 hour.



b. Aged 10 hours.



c. Aged 100 hours.



d. Aged 1000 hours.

Fig. 3. Electron micrographs of Inconel-X specimens solution-treated 4 hours at 2050°F and aged at 1600°F. X 12,000.

The heaviest grain-boundary precipitation occurred at 1400°F. Definite precipitation was evident at the grain boundaries of the specimen aged one hour at this temperature. With longer aging the precipitate became rapidly heavier, and in the specimens aged 100 and 1000 hours appeared to have agglomerated into separated particles. At 1200°F, the grain-boundary precipitation was similar to that at 1400°F, but generally much lighter. The grain-boundary precipitation at 1600°F, however, appeared to differ from that at the lower temperatures. Only a fine deposit was observed in the specimens aged 1 and 10 hours at this temperature. In addition, there were depleted bands along the grain boundaries in these specimens which were devoid of particles of the matrix precipitate. Such bands were not observed in any of the specimens aged at 1200° or 1400°F. In the specimens aged 100 and 1000 hours at 1600°F, the particles of the grain-boundary precipitate were difficult to distinguish from those of the matrix precipitate, and it was generally difficult to locate well-defined grain boundaries.

The matrix precipitate produced during aging at 1200°F consisted of a very fine dispersion of particles which were uniformly distributed throughout the matrix grains. In the specimens aged 1000 and 100 hours, these particles were estimated to average about 150Å and 100Å in diameter, respectively. In the specimen aged 10 hours they appeared to be still smaller and were often very difficult to recognize. It was estimated that the average separation of these particles did not exceed 150 - 200Å in any of these specimens. Aging at 1400° and 1600°F produced more rapid precipitation and led to the development of larger and more widely separated particles. The distribution of the particles in the specimens aged 10 hours and 1 hour at these temperatures, respectively, was similar to that in the specimen aged 1000 hours at 1200°F, although the particles were perhaps somewhat larger on the average. Longer periods of aging at these temperatures produced a rapid increase in the size and separation of the particles. The particles in the specimen aged 1000 hours at 1400°F were markedly cubic in shape and were seen in many micrographs to be arranged in square arrays in the surfaces. The largest of these particles were 1500 - 2000Å along the edge. The particles in the specimens aged at 1600°F developed spheroidal shapes and were not arranged along preferred directions in the matrix to the extent observed at 1400°F. The largest of these particles exceeded 10,000Å in diameter in the specimen aged 1000 hours at 1600°F.

MINOR PHASES

X-ray diffraction patterns were obtained from the residues separated from the unaged specimen and from the specimens aged 1 and 1000 hours at 1200°, 1400°, and 1600°F. The patterns from all of these residues consisted of two sets of face-centered cubic diffraction lines having the "d" values and relative intensities given in Table I. As shown in the table, these patterns agree very well with those reported for NbN and TiN. In all of the patterns the NbN diffraction lines were considerably stronger

than those of TiN, suggesting that the alloy generally contains somewhat more of the NbN than of the TiN phase. There were no detectable variations in the d values or the relative intensities of the diffraction lines to indicate changes in the compositions or relative amounts of the TiN and NbN phases as a function of the aging of the alloy.

TABLE I
COMPARISON OF X-RAY DIFFRACTION DATA FROM RESIDUES
SEPARATED FROM INCONEL-X SPECIMENS WITH DIFFRACTION
DATA FOR TiN AND NbN

| Inconel-X Residues | | TiN Pattern* | | NbN Pattern* | |
|--------------------|----|--------------|---|--------------|----|
| d | I | d | I | d | I |
| 2.53 | s | | | 2.53 | s |
| 2.43 | m | 2.44 | m | | |
| 2.19 | s | | | 2.19 | s |
| 2.11 | m | 2.11 | s | | |
| 1.55 | ms | | | 1.55 | ms |
| 1.49 | w | 1.49 | s | | |
| 1.32 | m | | | 1.32 | m |
| 1.27 | vw | 1.28 | m | 1.27 | w |

*The d values for these patterns were calculated from the lattice parameter values of 4.38Å and 4.23Å reported for NbN and TiN, respectively.⁸ The relative intensities are based on the data reported by Rosenbaum.⁷

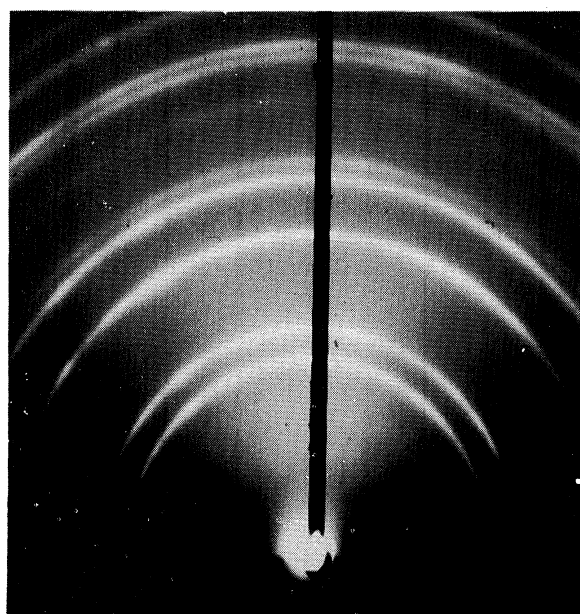
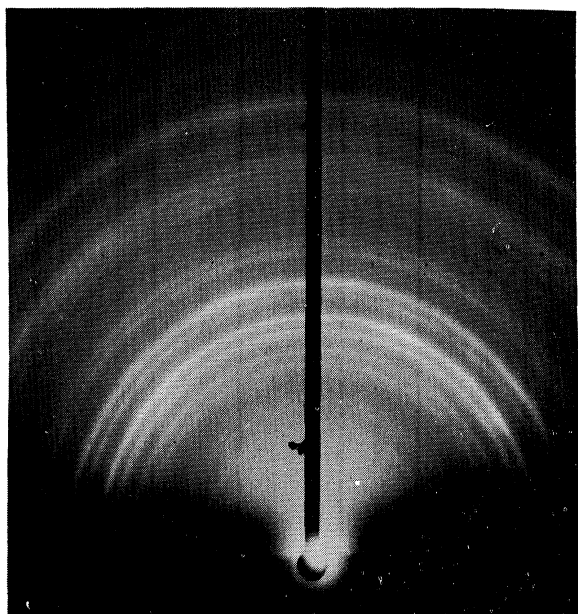
Repeated electron diffraction examinations were made of all of the specimens, using each of the etching reagents described above in preparing each specimen for examination. Very good electron diffraction patterns were obtained from all specimens. The predominant rings in the patterns from the specimens aged at 1200° and 1400°F corresponded to an M₂₃C₆-type complex carbide. In addition, weaker rings corresponding to the NbN and matrix phases were present in most of the patterns and some contained very weak rings which corresponded to TiN. A typical pattern from the specimen aged 1000 hours at 1400°F is reproduced in Fig. 4a, and the data from this pattern are compared with data for these different phases in Table II. The patterns obtained from the unaged specimen and from the specimens aged at 1600°F consisted predominantly of diffraction rings corresponding to the NbN and matrix phases of the alloy. In a few patterns very weak rings corresponding to TiN were observed, but none of these patterns contained rings corresponding to the M₂₃C₆ carbide. A pattern from the specimen aged 1000 hours at 1600°F is reproduced in Fig. 4b,

TABLE II

COMPARISON OF ELECTRON DIFFRACTION DATA FROM AGED
INCONEL-X SPECIMENS WITH DIFFRACTION DATA* FOR $M_{23}C_6$,
NbN, AND THE MATRIX PHASE

| Specimen Aged 1000 hr at 1400°F | | Specimen Aged 1000 hr at 1600°F | | $M_{23}C_6$ Pattern | | NbN Pattern | | Matrix Pattern | |
|------------------------------------|----|------------------------------------|---|------------------------|----|----------------|----|-------------------|----|
| d | I | d | I | d | I | d | I | d | I |
| 6.3 | w | | | 6.13 | | | | | |
| 5.3 | w | | | 5.32 | | | | | |
| 3.8 | w | | | 3.76 | | | | | |
| 3.2 | w | | | 3.21 | vw | | | | |
| 3.1 | w | | | 3.07 | vw | | | | |
| | | | | 2.66 | w | | | | |
| 2.54 | m | 2.53 | s | | | 2.53 | s | | |
| | | | | 2.43 | vw | | | | |
| 2.37 | ms | | | 2.38 | s | | | | |
| 2.18 | s | 2.19 | s | 2.17 | s | 2.19 | s | | |
| 2.06 | s | 2.05 | m | 2.04 | s | | | 2.06 | s |
| 1.88 | ms | | | 1.88 | m | | | | |
| 1.79 | ms | 1.79 | m | 1.79 | s | | | 1.78 | ms |
| | | | | 1.77 | m | | | | |
| 1.69 | w | | | 1.68 | vw | | | | |
| | | | | 1.62 | vw | | | | |
| 1.60 | w | | | 1.60 | vw | | | | |
| 1.55 | m | 1.56 | m | 1.53 | | 1.55 | ms | | |
| 1.48 | w | | | 1.49 | vw | | | | |
| | | | | 1.48 | vw | | | | |
| 1.42 | w | | | 1.42 | | | | | |
| 1.38 | w | | | 1.38 | vw | | | | |
| 1.33 | m | 1.34 | m | 1.33 | v | 1.32 | m | | |
| | | | | 1.30 | | | | | |
| 1.28 | w | | | 1.29 | | | | | |
| 1.26 | m | 1.26 | m | 1.26 | s | 1.27 | w | 1.26 | m |
| 1.23 | w | | | 1.23 | w | | | | |

*The d values given here for the $M_{23}C_6$ pattern include all permissible values for a face-centered cubic structure and were calculated for a lattice parameter of 10.63Å. This value and the intensity values given are based on data published by Rosenbaum.⁷ The NbN data are described in Table I. The matrix data were obtained by x-ray diffraction, using Cu- $K\alpha$ radiation.



a. Aged 1000 hours at 1400°F

b. Aged 1000 hours at 1600°F

Fig. 4. Electron diffraction patterns from aged specimens of Inconel-X alloy.

and the data for this pattern are included in Table II. The agreement between the electron diffraction data and the x-ray data reported for these different phases is very good as shown in the table.

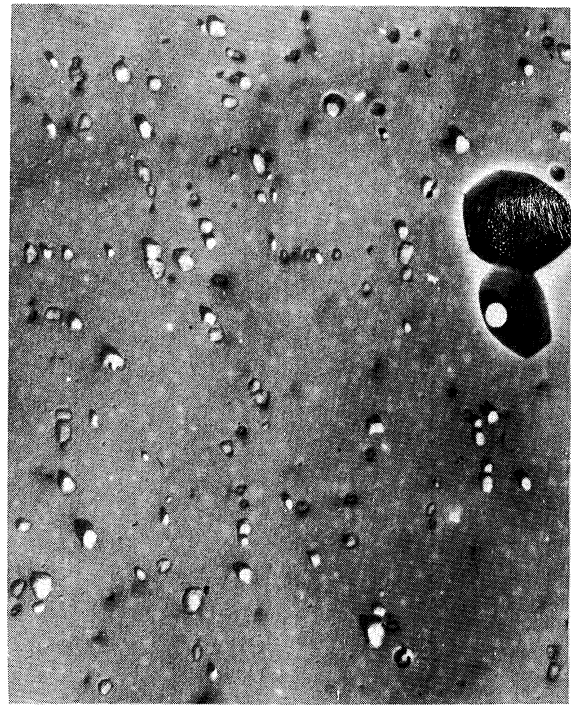
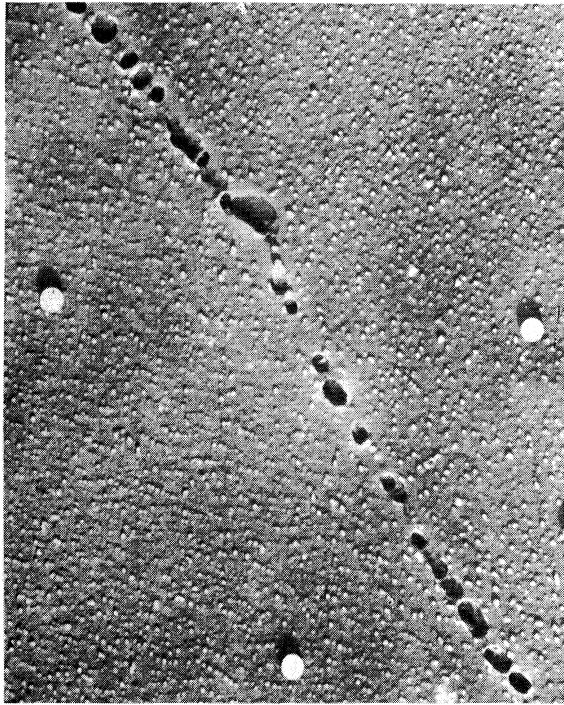
The identification of the $M_{23}C_6$ phase and the observed variations in its occurrence in the alloy, depending on the aging temperature, are considered to be reliable, for the electron diffraction patterns were of excellent quality and were readily reproduced even when different etchants were used in preparing the specimen surfaces. It is interesting that these patterns generally contained more diffraction lines of the $M_{23}C_6$ carbide than are usually reported from x-ray diffraction studies, particularly at high d values. Lattice parameters calculated from several patterns from each specimen all were in the range of 10.6 to 10.7Å. There were no indications of an increase in the lattice parameter with increasing periods of aging to indicate a transformation of the $M_{23}C_6$ carbide to an M_6C carbide as was observed in the 16-25-6 alloy.¹⁴

The electron diffraction data on the NbN phase were also very good in all cases. The data on the TiN phase were somewhat less satisfactory, however, since the reflections from this phase were generally very weak or were missing completely from the electron diffraction patterns. The

identification of this phase is therefore more reliably based on the x-ray results described above. The reflections in the electron diffraction patterns attributed to the matrix phase in Table II could equally well have been produced by the intermetallic γ' phase, for the structures of these phases are so similar that they would be difficult to distinguish on the basis of electron diffraction data, where the usual limit of accuracy is about $\pm 1\%$. Consideration of the electron micrographs supports this possibility, particularly in the case of patterns from surfaces of aged specimens which were prepared by etching with reagent B. The micrographs of Figs. 1, 2, and 3 show that these specimens contain numerous small precipitate particles which protrude above the matrix phase in the etched surfaces. Furthermore, the matrix phase exposed in these surfaces is very smooth. In studies of other alloys,¹⁰ using surfaces of similar character, diffraction rings of the matrix phase were seldom obtained. There is therefore no reason to expect strong matrix contributions in the Inconel-X specimens, particularly in view of the large number of exposed minor-phase particles.

The x-ray diffraction studies on the bulk specimens provided a more direct identification of the γ' phase. Patterns obtained by back reflection from surfaces of the specimens aged 1000 hours at 1200°, 1400°, and 1600°F contained weak diffraction lines adjacent to the strong matrix lines at positions corresponding to a phase having a cubic unit cell about 0.5% larger than that of the matrix. These results are similar to those obtained by Nordheim and Grant⁵ in studies of the γ' phase of other nickel-chromium alloys of similar titanium and aluminum content. One very interesting x-ray diffraction pattern was obtained from a very fine needle which was cut from the specimen aged 1000 hours at 1400°F. This pattern was recorded in a Debye-Scherrer camera and was very spotty due to the limited number of grains exposed to the x-ray beam; however, adjacent to each strong matrix spot was a weaker γ' spot which in all cases was located on a radius joining the matrix spot and the center of the pattern. This could occur only if the γ' spots were from the same grains as the corresponding matrix spots, and if the two phases had the same crystallographic orientation in the grains. An orientation of this type is to be expected since both phases have face-centered cubic unit cells of very nearly the same size.

The selective etching procedure for differentiating the carbide and γ' particles was applied to all of the specimens. The electron micrographs showed that the selective etching action was entirely on the particles which precipitated within the matrix grains during aging. The large particles observed to be present after solution treatment and the precipitates which formed in the grain boundaries during aging were not attacked. This is illustrated by the electron micrographs of Fig. 5. Additional examples of the application of this etching procedure have been presented elsewhere.¹²



a. Aged 100 hours at 1400°F

b. Aged 100 hours at 1600°F

Fig. 5. Electron micrographs of aged specimens of Inconel-X alloy selectively etched to distinguish γ' particles from carbide and nitride particles. X 12,000.

DISCUSSION

Four different minor phases have been identified in the aged specimens of the Inconel-X alloy: titanium nitride, niobium nitride, an $M_{23}C_6$ -type complex carbide, and the intermetallic γ' phase. The titanium and niobium nitrides were found in all of the aged specimens and in the solution-treated alloy. The $M_{23}C_6$ phase occurred only in the specimens aged at 1200° and 1400°F. The intermetallic γ' phase was found to develop at all temperatures of aging. These results are summarized in Table III.

Comparison of the diffraction results and the observations on the microstructure of the alloy make it possible to identify the different types of precipitates which were observed, and thereby to obtain considerable information on the distribution of the different phases within the alloy. The large spheroidal and angular particles can be identified as particles of the titanium and niobium nitrides from the results on the solution-treated, unaged specimen, inasmuch as these were the only precipitate particles and the only minor phases found in this specimen. That

TABLE III

MINOR PHASES IDENTIFIED IN AGED*
SPECIMENS OF INCONEL-X ALLOY

| Time of Aging, hr | Temperature of Aging | | |
|-------------------|---|---|----------------|
| | 1200°F | 1400°F | 1600°F |
| 1 | | NbN, TiN M ₂₃ C ₆ , γ' | NbN, TiN γ' |
| 10 | NbN, TiN M ₂₃ C ₆ | NbN, TiN M ₂₃ C ₆ , γ' | NbN, TiN γ' |
| 100 | NbN, TiN M ₂₃ C ₆ , γ' | NbN, TiN M ₂₃ C ₆ , γ' | NbN, TiN γ' |
| 1000 | NbN, TiN M ₂₃ C ₆ , γ' | NbN, TiN M ₂₃ C ₆ , γ' | NbN, TiN γ' |

*All specimens were solution-treated 4 hours at 2050°F prior to aging. Only TiN and NbN were identified in the alloy in the solution-treated condition.

these particles persist under all of the aging conditions is not surprising in view of their apparent stability at the solution-treating temperature. The fact that they appear to increase slightly in size with long periods of aging suggests that they account for virtually all of the niobium and titanium nitride phases which precipitate. The electron microscopic studies using the selective etching procedure show that the numerous particles which precipitate within the matrix grains of the alloy during aging are the intermetallic γ' phase. This is consistent with the x-ray diffraction results from the bulk specimens, for a very large number of particles would be required to give x-ray reflections from this phase under the experimental conditions used. Finally, the material which precipitates at the grain boundaries during aging appears to consist predominantly of the complex M₂₃C₆ carbide, for electron diffraction patterns of this phase were obtained only from the specimens aged at 1200° and 1400°F which contain heavy grain-boundary precipitation. The light precipitation in the grain boundaries of the specimens aged for a short period at 1600°F probably consists of the TiN and NbN phases. The absence of a well-defined grain-boundary precipitate in the specimens aged for long periods at this temperature can be readily accounted for by redistribution of this material to form larger particles or to add to the existing particles.

It is of particular interest to consider these observations on the minor phases in relation to the high-temperature properties of the

alloy. In their studies of the effects of high-temperature aging on the metallurgical properties of the alloy, Frey, Freeman, and White¹ found variations in the hardness, the creep strength at high stresses (100,000 psi, measured at 1200°F), the rupture strength at low stresses (60,000 psi, measured at 1200°F), and the 2% yield strength (measured at 1200°F). Aging at 1200°F produced a gradual, monotonic increase in these properties from the initially low values of the solution-treated alloy to very high values for specimens aged 1000 hours. Aging at 1400° and 1600°F produced more rapid initial increases in these properties with much shorter periods of aging; however, maximum values were reached after 10 to 100, and 1 to 10 hours of aging, respectively, and further aging caused gradual decreases. For equal periods of aging, the values at 1400°F were generally higher than those at 1600°F, and the maximum values at these temperatures did not exceed those reached with long periods of aging at 1200°F. These general relationships are represented by the curves of Fig. 6.

X-ray diffraction studies showed corresponding variations in the internal strain of the alloy, and from consideration of the character of this strain it was concluded that the above variations in the metallurgical properties were controlled by the precipitation of minor-phase particles within the matrix grains of the alloy with an average spacing between particles conforming to that of "critical dispersion" as defined by Mott and Nabarro.¹⁵ This spacing was calculated to be of the order of 700Å.

From the results of the present investigation it is apparent that the minor phase in question is the intermetallic γ' phase, since this is the principal minor phase to develop in the matrix grains of the alloy. Furthermore, it is possible to obtain a qualitative correlation between the microstructure of the alloy and the variations in the "matrix-controlled" metallurgical properties. Upon comparison of the electron micrographs of Figs. 1, 2, and 3 with the curves of Fig. 6 it is evident that at all temperatures the periods of aging required to cause an increase in the "matrix-controlled" properties are about the same as those required to produce detectable γ' particles in the alloy, and that the properties continue to increase during the early stages in the development of these particles. During these periods, the particles are very small, usually averaging only a few hundred Angstrom units in diameter, and their average separation is in the general range of the value calculated by Frey, Freeman, and White¹ for the condition of critical dispersion. It is also probable that during these periods the γ' particles are coherent with the matrix phase. This is suggested by their very small size, by the similarity in the size and type of the crystallographic unit cells of the γ' and matrix phases, and by the high internal strain of the alloy during these periods. The maxima in the "matrix-controlled" properties during aging at 1400° and 1600°F occur as the γ' particles begin to coalesce and to depart from the condition of critical dispersion. Continued aging produces continuing growth of the particles and continuing decreases in the properties. The fact that the particles take on well-defined, characteristic shapes, and that the internal strain of the alloy decreases during

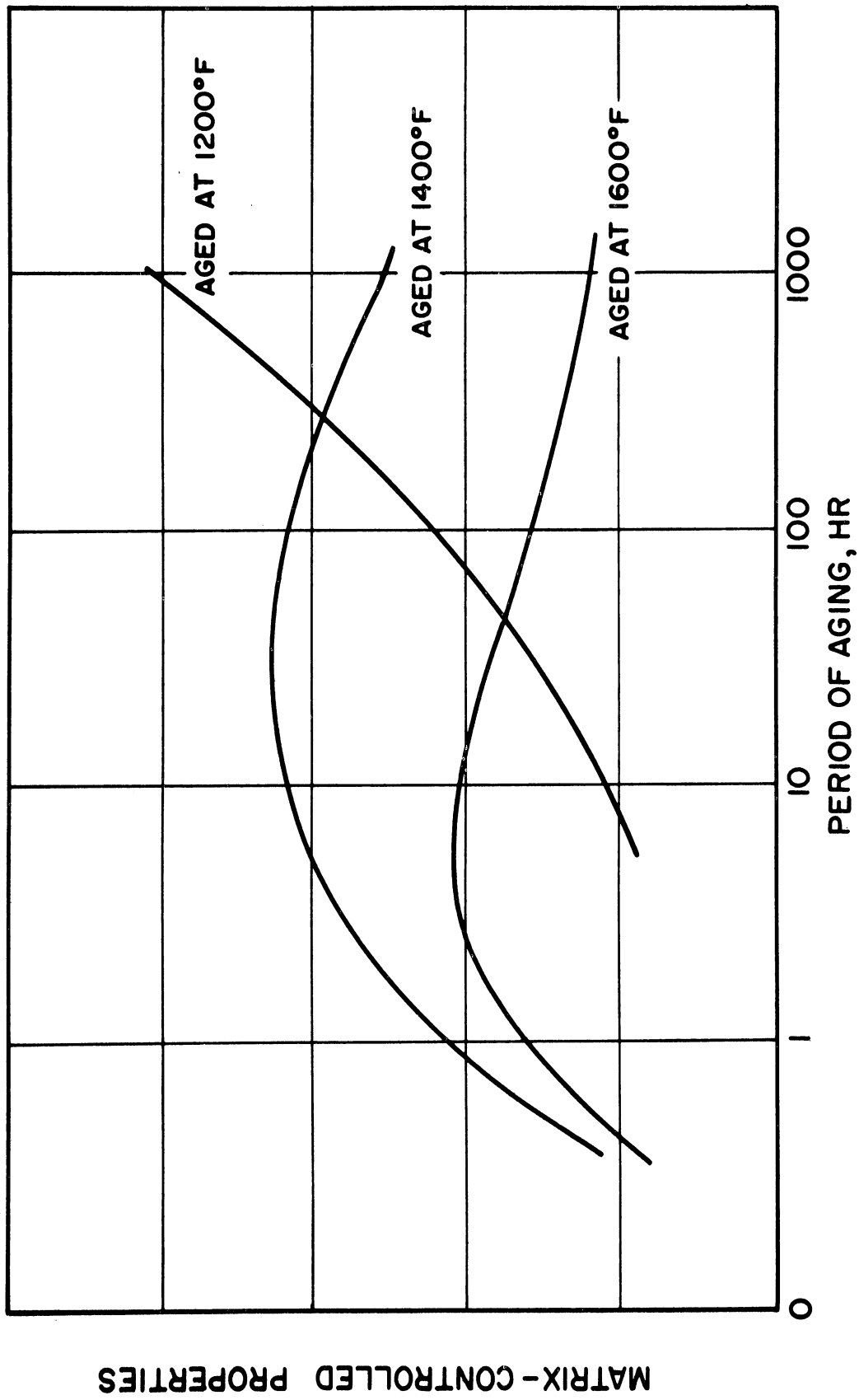


Fig. 6. Variation of matrix-controlled properties of Inconel-X alloy with aging (schematic generalization of data of Frey, Freeman, and White¹ on hardness, rupture strength, creep strength, and yield strength).

these periods, suggests that the γ' particles also become incoherent.

These conclusions attributing the age hardening and strengthening of the Inconel-X alloy to the precipitation of the γ' phase are consistent with and lend support to the conclusions of Hignett⁶ and Nordheim and Grant⁵ concerning the role of this phase in determining the properties of other similar nickel-base alloys. In addition, the correlation of the size and distribution of the γ' particles with the variations in the metallurgical properties of the alloy provides direct confirmation of the conclusions of Frey, Freeman, and White¹ concerning the dependence of the properties on the dispersion of the precipitate particles in the matrix of the alloy. It is of particular interest that when the γ' phase is exerting maximum influence on the properties of the alloy, it is in the form of particles which are sufficiently well developed to be recognizable by electron microscopy. This is contrary to the opinion sometimes expressed that the maximum precipitation hardening of the nickel-base alloys occurs during the nucleation stage of the precipitation process prior to the development of well-defined particles of the γ' phase. This is of considerable importance, for it suggests that many fundamental aspects of the precipitation phenomena in these alloys can be investigated by electron microscopy.

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