RELATIONSHIP BETWEEN SMALL
ANGLE DISLOCATION BOUNDARIES
AND CREEP

FIFTEENTH TECHNICAL REPORT

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JUNE 1954

SERIES NO. 28 ISSUE NO. 23
CONTRACT NO. AT-11-1-GEN-10, PROJECT I

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W.O. 44455

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Fifteenth Technical Report

under

U. S. Atomic Energy Commission

Contract AT-11-1-Gen-10, Project 1

June 1954

Series No. 28

Issue No. 23

Introduction:

A metal subjected to a constant stress at elevated temperatures continuously deforms or "creeps". The creep phenomenon has been investigated extensively under many varying conditions, but the atomic mechanisms involved apparently are so complicated that no satisfactory theory has been developed. It is now well established that during creep the individual grains of a metal "fragment" into subgrains bounded by arrays of dislocations formed as a result of non-homogeneous deformation within the grains^(1, 2, 3).

It has been demonstrated that deformation previous to creep markedly affects the creep resistance of pure aluminum^(4, 5), pure nickel, and nickel alloys⁽⁶⁾. The introduction of subboundaries causes a decrease in creep rate and (at least in the case of pure nickel and nickel alloys) changes the shape of the creep curve⁽⁶⁾. The creep studies reported in the present paper were undertaken to determine the nature, density and effect of small angle boundaries.

Quantitative relationships between subboundary density and room temperature tensile strength have been established and reported elsewhere⁽⁷⁾. This work showed that the yield strength of pure nickel is an approximately linear function of the number of small angle boundaries per grain. Also Washburn⁽⁸⁾ has studied the dependence of yield strength on the orientation of the subgrains. In his experiments the yield stress in simple shear of an initially very nearly perfect zinc single crystal was increased 25% by the presence of a pair of 0.05° boundaries composed of simple edge dislocations. When the boundary angle was doubled, the accompanying increment of strengthening was less; the rate of strengthening decreased with

increasing angle and a saturation value of about 50% total increase in strength appeared to be approached for boundaries somewhat over 1° . Similar results were obtained for complex arrays of tilt boundaries and for screw dislocation arrays. Thus, it has been established that small angle boundaries are very effective as barriers to further deformation. The magnitude of the effectiveness depends upon both the number and the boundary angle. A third factor of importance is the nature of the boundary.

The present authors⁽⁷⁾ have shown that diffuse subboundaries are produced simultaneously with room temperature tensile deformation in nickel. Polycrystalline aggregates containing such diffuse small angle boundaries have a maximum strength for a given subboundary density. The large increases in strength accompanying the presence of diffuse subboundaries in such specimens suggest that many distributed dislocations are trapped at internal barriers causing significant hardening. A suitable thermal treatment causes these diffuse subboundaries to become sharp; a redistribution of dislocations occurs during this treatment, with some of them being annihilated and others forming dislocation walls. The metal is then softer than it was for an equivalent array of diffuse subboundaries. Individual boundaries in simple shear become better barriers the more perfectly the dislocations are aligned⁽⁸⁾. However, a complicated pattern of tangled dislocations, causing intensive local bending, is even more effective in hardening than the arrangement of dislocations in a highly polygonized metal. Thus the nature and density of small angle dislocation boundaries are known to influence the strength both in simple shear and in tension. The results of investigations carried out to determine (1) the nature and density of small angle

boundaries developed during creep, and (2) the effect of controlled densities of pre-existent subboundaries on creep, are presented herein.

Experimental Procedure:

High purity nickel (99.95%) was refined, cast, forged and swaged in the authors' laboratories and machined into specimens with two reduced sections of different lengths in tandem as previously described⁽⁹⁾. All specimens were annealed one-half hour at 1150° C to remove cold work and to produce an appropriate grain size (~ 0.5 mm in diameter) for x-ray microscopic examination of the substructure. An x-ray microscope⁽¹⁰⁾ was designed to have an average resolving power of about four microns with a maximum of a fraction of a micron. The Bragg reflections were recorded on fine grain photographic plates which were magnified up to 250 times. With this procedure, angular differences between subgrains of just a few minutes were detectable.

Controlled amounts of tensile strain were given specimens at room temperature; these were then recovered for one hour at 800° C. Following this "prestrain" treatment the bars were tested at 700° C and 5750 psi in single specimen creep units which were constructed to hold the stress to within $\pm \frac{1}{2}\%$ and the temperature within $\pm \frac{1}{2}^\circ \text{C}$ ⁽⁹⁾. The creep tests were halted at strains of 1% or $2\frac{1}{2}\%$ and the specimens rapidly cooled to room temperature by means of a helium jet. One-half of these specimens was then submitted to tension tests and the duplicate series was used for substructure studies. Specimens prepared for x-ray microscopic examination were carefully cross-sectioned in the center of the long gage sections, polished, and then deeply

etched to avoid spurious effects resulting from the sectioning. It was thus possible to study the relationships between small angle boundary density, the creep strength and room temperature tensile properties before and after creep.

Experimental Results:

As has been previously shown^(7, 8, 11), the introduction of small angle dislocation boundaries by local plastic bending and appropriate heating greatly increases the yield strength of nickel and other metals. This is illustrated in Fig. 1⁽⁷⁾ which shows the almost linear relationship between flow stress and substructure density for pure nickel. X-ray micrographs of typical grains from specimens given a recovery treatment following varying prestrains which produce different strengths are shown in Fig. 2. The specimen with the least strength (zero percent prestrain) was characterized by an almost complete absence of subboundaries as shown in Fig. 2a.

The strain rates at the beginning of creep (Fig. 3) varied from very large values to nearly zero, depending on the initial substructure. Accordingly creep curves were produced for which the strain rates were continuously decreasing, constant, or progressively increasing. These tests were interrupted at 1% or 2½% creep strain and the specimens tensile tested at room temperature. Fig. 5 shows that all had essentially the same strength, independent of creep rate, initial subboundary density and amount of creep strain. This strength level corresponded to that of a specimen which had been prestrained at room temperature to about 2½% and recovered, containing an average of 5 or 6 subboundaries per grain as shown by the data of Fig. 1.

Several of the specimens were prestrained sufficiently to induce partial recrystallization during the heat treatment. A typical stress-strain curve of a partially recrystallized specimen is shown in Fig. 6 and creep curves of similar specimens are given in Fig. 7. The strain rate during creep decreases more rapidly for the recrystallized nickel than for unrecrystallized material even though the initial rates are comparable. The tensile strength, however, after a creep strain of $2\frac{1}{2}\%$ (Fig. 5) also corresponded to that found for specimens which had acquired an average of 5 or 6 small angle boundaries per grain by pulling in tension and subsequently heating.

The substructure investigations yielded astonishing results. The distribution of small angle boundaries in one typical grain of a specimen without prestrain which was crept to $2\frac{1}{2}\%$ is shown in Fig. 8a. A very large number of boundaries having angles of just a few minutes are visible. The subboundary distribution in another grain from the same specimen is reproduced in Fig. 8b. Although the latter is not typical in that it also contains a relatively large subboundary*, it is apparent that the density of very small angle subboundaries is also high. Thus, a creep strain of $2\frac{1}{2}\%$ produces an average number of small angle subboundaries which is so large that they cannot be counted, yet only an average of 5 or 6 subboundaries per grain are created by tensile straining a similar specimen at room temperature (cf. Figs. 1 and 2). On the other hand, the room temperature tensile strength produced by either straining in creep to $2\frac{1}{2}\%$ at 700°C , or tensile straining the same amount at room temperature (followed by an 800°C recovery treatment) are the same.

* or a relatively wide subboundary. The distinction between a wide subboundary and a large angle one may not be made with simple x-ray microscopic techniques.

Specimens which had been prestrained 2%, 3 1/3% and 5%, respectively, and then crept to either 1% or 2 1/2% yielded a subboundary distribution similar to the no-prestrain specimens. No differences could be observed either as a function of prestrain in tension or creep strain. As an example, Fig. 9a is an x-ray micrograph of a grain from a specimen prestrained 3 1/3% in tension and 2 1/2% in creep while Fig. 9b is from a similar specimen interrupted after only 1% creep strain. The lack of dependence of subboundary density on the amount of creep strain is further verified by the density observed in a specimen with no prestrain which was crept 7.7% to fracture, as shown in Fig. 10. Both the density and the nature of the subboundaries were very similar to those observed in Fig. 8 for a specimen crept to only 2 1/2%.

Specimens which were prestrained to such a large elongation that they recrystallized either before or during creep also exhibited a high density of small angle boundaries. Fig. 11 shows not only very small angle boundaries which are similar to those already observed, but also "diffuse" subboundaries. In spite of the fact that the nature of the substructure in the recrystallized specimens differed from those with less prestrain, all the creep specimens had a very high density of subboundaries and the room temperature tensile properties of all these specimens corresponded to specimens having a subboundary density of five or six per grain produced by straining at room temperature.

In order to observe the effect of additional room temperature deformation on the multitude of very small angle subboundaries developed during creep, three of the specimens responsible for the curves in Fig. 3 were examined further. They were prestrained 0%,

3 1/3% and 5% at room temperature, recovered as usual, crept 2 1/2%, 1% and 1% respectively, and then all deformed an additional 2% at room temperature. Finally these specimens were recovered again at 800° C and examined by x-ray microscopy. The multitude of small angle boundaries had disappeared and there remained subboundaries similar in both density and nature to those produced by simply prestraining annealed material approximately 4% at room temperature and recovering it.

Further x-ray investigations were made to determine statistically the average range of misorientation within grains of the various specimens. The usual back reflection technique was employed and the angular spread within each of ten to twenty grains was averaged for each specimen. The results are recorded in Table I.

Table I

Tensile, %	Total Strain		Range of Orientation per Grain	
	Creep, %		Average	Maximum
0	2 1/2		6°	10°
2	0		5°	14°
6 3/4	2 1/2		17°	38°
7	0		15°	37°

The total spread in orientations within a grain may be interpreted as the sum of the subboundary angles per grain as measured along some arbitrary direction. This value increases with the total strain. The sum of the subboundary angles in the specimens pulled to the higher strains is at least 38° in some grains.

Discussion:

The shape of the creep curve may be made to vary as a function of the initial density of small angle boundaries as shown in Figs. 3 and 7; thus a new research tool is available for investigating the causes of the progressive change in creep rate during a test. "Primary" creep can even be eliminated by introducing small angle boundaries of a certain nature and in the proper density. Since the mean subboundary density and nature may now be readily controlled and detected⁽⁷⁾, creep deformation can be analyzed in a new light.

Creep resistance increases with increasing density of sharp small angle boundaries introduced by room temperature deformation and subsequent recovery, unless, of course, recrystallization occurs. Boundaries introduced by prestraining and heating do not have the same effect on creep rate as those developed during creep; the "artificially" produced subboundaries have a different structure and cause much more prolonged periods of low creep rate (cf. Fig. 3 and 4). The effect of varying natures of subboundaries, as controlled by recovery treatment, remains to be determined.

Substructure produced by creep is very different from that developed by an equal amount of room temperature deformation plus recovery (cf. Figs. 8 and 2b). Not only is the subboundary density very much higher in the case of creep, but also the creep process produces a unique type of structure containing a very large number of extremely small angle boundaries which are closely spaced; these subboundaries are usually parallel and of short extent. The sum of the subboundary angles, as measured along some arbitrary direction, is the same for equal quantities of creep or tensile strains (according

to the x-ray back reflection data in Table I). One would, therefore, be led to conclude from the back reflection experiments alone that the substructure of crystallites subjected to either creep or tensile strain was the same, since no differences were observed between the films from such specimens. It is clear that extreme caution must be exercised in interpreting x-ray back reflection diagrams and that the back reflection technique is not a discriminating method for investigating substructure.

The unique characteristic of the substructure developed during creep, as compared with that produced by room temperature deformation plus recovery, is the large number of extremely small angle parallel subboundaries. The reason for the difference must be understood before a theory of creep can be developed.

Plastic deformation under any circumstances results in the piling up of dislocations at barriers which are already present within a crystal. Such a barrier may be, for example, a screw dislocation cutting across the active slip plane or a sessile dislocation. If the conditions under which deformation occurs are suitable, migration of piled-up dislocations perpendicular to the slip plane may occur by diffusion. This climb process requires the solution or precipitation of vacancies at the centers of the dislocations. The controlling factor is the rate at which atomic "jumps" can take place. If the jump frequency is too low, appreciable climb cannot occur. According to the Eyring rate theory, the jump frequency may be written

$$(1) \quad \nu = \frac{kT}{h} \exp (-Q/RT)$$

where Q is the activation energy for the process. The activation energy for vacancy migration is thought to be approximately one-half of the activation energy for self-diffusion⁽¹²⁾. This value has not been measured for nickel, but the activation energy for creep, which is generally the same (cf. Table II in (13)) as that for self-diffusion, is about 65 kcal⁽⁹⁾. If one-half this value is substituted in equation (1), the calculated jump frequency is $2 \cdot 10^6$ per second at 700° C, the temperature at which the creep tests were conducted.

Experimentally it was observed that large numbers of very small angle boundaries formed after only a few minutes of creep (e.g. Fig. 8b). Calculations indicate that the time necessary for a boundary having an angle of 10 minutes to development (if atomic jumps can occur at the rate of $2 \cdot 10^6$ per second) is only a few seconds; such a boundary is composed of dislocations about 333 atomic planes apart. If the boundary is 0.1 mm long, then there are 1000 dislocations in the boundary or if the boundary develops symmetrically with respect to the slip plane, 500 on each side of the active slip plane. Thus about $4 \cdot 10^7$ jumps are required in each direction to build a 10 minute boundary, a process requiring only about 20 seconds. It thus appears that high temperature deformation provides favorable conditions for the occurrence of climb during the deformation process and that creep deformation therein differs from plastic flow at low temperatures.

Eventually a boundary may grow to such a large extent perpendicular to the active slip plane that further lengthening is less likely than the starting of a new boundary. In the example taken from Fig. 8a, as much time is required to add only two dislocations to a 10 minute boundary 0.1 mm long as is required to build a new one of the same

angle containing ~ 30 dislocations. The local stress, caused by the piled-up dislocations on the slip planes, remains substantially constant during the formation of boundaries by climb, since as soon as a certain number of dislocations leaves the pile, the Frank-Read sources may generate replacements. The length of the Frank-Read source would be a controlling factor in the replacement process.

All the dislocations in the slip plane have a tendency to leave the region of high local stress, which they can do by diffusing up or down from the slip plane. When a dislocation somewhere back in the line moves into a nearby slip plane, it is repelled by the already growing boundary and travels back toward the Frank-Read source. As it glides back toward the source in a plane either above or below the active slip plane, the dislocation falls successively into minimum potential energy positions over each dislocation still in the slip plane (see Fig. 12). Eventually, when the force of repulsion from the already growing boundary is no longer sufficient to drive it further away, the dislocation will be trapped in one of the energy wells. By this process short new boundaries can develop parallel to each other.

In the experiments, the substructure configuration as developed by creep was "frozen in" the specimens by reducing the temperature rapidly under load. It was observed that independent of the initial substructure before creep and of the sum of subboundary angles within the grains after creep, all specimens corresponded to that characteristic of a prestrained-recovered ($2\frac{1}{2}\%$) specimen containing an average of only five or six sharp subboundaries per grain (cf. Figs. 5 and 1). The multitude of small angle boundaries disappeared

after tensile testing and only a relatively small number of large ones remained. The number remaining after this further strain of 2% was approximately the same as a specimen prestrained about 4%. The conditions of creep, i.e. the temperature and stress levels, appeared to have a dominant influence on the properties of the substructure. The reasons for this conclusion are (1) after creep a common strength is reached independently of the strength level before creep; (2) the substructure after tensile testing and recovering is the same for all specimens and is like that produced by recovering a specimen tensile prestrained about 4%.

These results may be understood as follows: as the stress in the tensile test is increased above the creep stress, dislocations are again generated and begin to pile up at the tiny boundaries introduced by creep. When the stress reaches a certain value (in the present experiments about 10^4 psi or nearly twice the creep stress), many of the barriers developed during creep cease to be effective barriers. Both theory and experiment indicate that many of the unique very small subboundaries introduced by the creep deformation may be moved by the applied stress and caused to coalesce into fewer but larger subboundaries. The material was much weaker than one would expect for a substructure full of subboundaries because the multitude of very small angle boundaries was annihilated during tensile testing and recovery. Such a merging of small angle boundaries has been observed frequently in single crystal work^(14, 15). A further conclusion is that some of the barriers which were effective in developing subboundaries at room temperature are permeable in a long time creep test at 700° C, since some specimens

were made weaker by creep, instead of stronger. It appears, then, that the temperature and stress level of the creep test uniquely determine not only the substructure developed, but also the room temperature tensile properties.

Summary:

1. The creep rate is a function of the initial quantity of small angle boundaries per grain.
2. The substructure produced by creep contains a multitude of very small angle boundaries as well as some larger ones of the type developed by cold-work and recovery. These very small angle boundaries are generally parallel and of small extent. Cold-work and recovery produces a different substructure free from a multitude of minute parallel subboundaries.
3. Calculations indicate that the unique part of the substructure developed during high temperature deformation is due to the climbing of dislocations originally piled up at barriers to slip.
4. The development during creep of short parallel boundaries can be caused by the interactions of local stress fields from (a) the climbing dislocations; (b) the dislocations in the boundary (or boundaries) already present; and (c) the piled-up array of dislocations on the active slip plane.
5. Specimens crept to varying amounts, with initially very different substructures, all developed the same substructure and had the same room temperature tensile strength. The substructures after these tensile tests and recovery were identical and were characterized by the absence of the very small angle boundaries.

ACKNOWLEDGMENT

The authors wish to express sincere appreciation to Mrs. Rosa Hansen, George Kilbourne and C. E. Pausa for their help in the testing program.

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FIG. 1 THE YIELD STRENGTH OF PURE NICKEL AS A FUNCTION OF SMALL ANGLE BOUNDARY DENSITY.

(a) AS ANNEALED

(b) 2% PRESTRAIN

(c) 4% PRESTRAIN

FIG. 2 X-RAY MICROGRAPHS OF INDIVIDUAL GRAINS
IN SPECIMENS STRAINED VARIOUS AMOUNTS
IN TENSION AND RECOVERED AT 800 °C

FIG. 3 - CREEP CURVES OF DIFFENT SHAPES RESULTING FROM VARIOUS INITIAL SMALL ANGLE BOUNDARY DENSITIES

FIG. 4 TYPICAL CREEP CURVE OF ANNEALED PURE NICKEL (700°C & 5750 psi)

FIG. 5 STRESS-STRAIN CURVES OF CREEP SPECIMENS INTERRUPTED AFTER CREEP STRAIN (ϵ_C) FOLLOWING A PRESTRAINING IN TENSION (ϵ_T) PLUS RECOVERY TREATMENT

FIG. 6 STRESS-STRAIN CURVE OF PARTIALLY RE-CRYSTALLIZED NICKEL CONTRASTED WITH UNRECRYSTALLIZED SPECIMENS

FIG. 7 CREEP CURVES OF PARTIALLY RECRYSTALLIZED NICKEL CONTRASTED WITH UNRECRYSTALLIZED SPECIMENS.

(a) A TYPICAL GRAIN. IN THE HIGHER MAGNIFICATION PHOTOGRAPH "CROSS-POLYGONIZATION" IS OBSERVABLE (UPPER RIGHT).

(b) A SOMEWHAT LESS TYPICAL GRAIN CONTAINING AN USUALLY WIDE OR LARGE ANGLE SUBBOUNDARY.

FIG. 8 X-RAY MICROGRAPHS FROM A SPECIMEN CREPT $2\frac{1}{2}\%$ SHOWING A VERY HIGH DENSITY OF EXTREMELY SMALL ANGLE BOUNDARIES. THE ENTIRE GRAIN IS REPRODUCED ON THE LEFT; ON THE RIGHT THERE APPEARS A MORE HIGHLY MAGNIFIED PORTION.

a)

(b)

FIG. 9 X-RAY MICROGRAPHS FROM SPECIMENS PRESTRAINED $3\frac{1}{3}\%$, RECOVERED AND CREPT (a) 1% AND (b) $2\frac{1}{2}\%$. THE SUBSTRUCTURE CANNOT BE DIFFERENTIATED. THE ENTIRE GRAIN IS REPRODUCED ON THE LEFT; ON THE RIGHT THERE APPEARS A MORE HIGHLY MAGNIFIED PORTION.

FIG. 10 X-RAY MICROGRAPHS FROM AN ANNEALED SPECIMEN CREPT $7 \frac{3}{4} \%$ TO FRACTURE. THE ENTIRE GRAIN IS REPRODUCED ON THE LEFT; ON THE RIGHT THERE APPEARS A MORE HIGHLY MAGNIFIED PORTION.

FIG. II X-RAY MICROGRAPHS FROM A PARTIALLY RECRYSTALLIZED SPECIMEN (PRESTRAINED $6\frac{3}{4}\%$) WHICH WAS CREPT $2\frac{1}{2}\%$. THE ENTIRE GRAIN IS REPRODUCED ON THE LEFT, ON THE RIGHT THERE APPEARS A MORE HIGHLY MAGNIFIED PORTION.

SCHEMATIC REPRESENTATION OF SUPERPOSITION OF POTENTIAL ENERGY WELLS, PRODUCED BY THE PILED-UP DISLOCATIONS, ON INTERACTION ENERGY OF AN EDGE BOUNDARY AND A SINGLE DISLOCATION.

A DISLOCATION WHICH MOVES OUT OF THE PILE-UP IS REPELLED BY THE BOUNDARY AND SO TRAVELS BACK IN THE DIRECTION OF THE SOURCE UNTIL IT FINDS A STABLE POSITION IN A POTENTIAL ENERGY WELL.

FIG. 12 MECHANISM OF PARALLEL SUBBOUNDARY FORMATION.

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