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SUBGRAIN REFINEMENT STRENGTHENING

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prepared by

Rodney Klundt, Bruno Walser, Yoshio Monma and Oleg D. Sherby

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Department of Materials Science and Engineering
Stanford University
Stanford, California 94305

ABSTRACT

During the past quarter (Jan. - March 1975) we have initiated mechanical properties studies on type 304 stainless steel and on a ferritic alloy, E-Brite 26-1. The purpose of these studies was to establish a sound data base from which the alloys specifically chosen for this program can be evaluated (namely, ferritic steel, precipitation hardening austenitic stainless steel and a nickel rich austenitic alloy).

GENERAL ACCOMPLISHMENTS

We received a visit from Mr. W. Bennett of U.S.E.R.D.A., Washington, D. C. and Dr. James Laidler of H.E.D.L., Hanford, Washington, on February 20, 1975. In the morning, Dr. Laidler presented a seminar on material performance and requirements in the LMFBR. He emphasized the problem of swelling and creep in the cladding and duct materials. It was pointed out that high nickel austenitic steels look very promising. Ferritic steels also appear promising as swelling resistant materials but were not particularly good for thermal creep. In the afternoon, we presented our general objectives and philosophy on the subgrain boundary strengthening program. An outline of our presentation is included in the appendix to this quarterly report. Extensive discussions on our planned work lead to the conclusion that it would be most useful to assess the possibility of subgrain boundary strengthening in two ferritic steels: EM 12 and HT 9. Both materials appear to exhibit negligible swelling using electron irradiation and observation in a 1 MeV electron microscope[1]. Rods of the two ferritic alloys were ordered from H.E.D.L.

In the following sections we describe some of our initial studies on 304 stainless steel and E-Brite 26-1. Tests were performed both in compression and in torsion as a means of developing subgrain structures in the warm temperature range of working. In addition, compression testing was employed as a means of studying the mechanical characteristics of subgrain-strengthened materials.

304 Stainless Steel

The stainless steel studied was prepared in the form of round circular cylinders, 1.22cm in diameter and 1.83cm long. The samples were machined from 304 stainless steel rods received in the as-cold-worked state. Initial tests were performed in compression at 900°C over a wide range of strain rates. These data are given in Figure 1 (the authors would like to acknowledge the assistance of Mr. Yasuo Watanabe who performed some of the original compression tests on 304 stainless steel). The data, in general, reveal a region of strain hardening followed either by a steady state flow stress or by some strain softening. This later effect was especially pronounced at low strain rates. Transmission electron microscopy studies revealed the formation of subgrains. The subgrain sizes developed were nearly identical in size to those obtained earlier in torsion tests on the same material(2). In addition, we noted the formation of twins at low strain rates but not at high strain rates. The strain softening observed is suggestive of deformation processes other than subgrain formation. It is possible that the original cold-worked state was not entirely eliminated during annealing and subsequent testing at 900°C. Thus, recovery could be occurring during deformation leading to strain softening. Tests are being performed at 1000 and 1100°C over a wide strain rate range (crosshead speeds from .0001 cm per minute to 1 cm per minute). Completely annealed material is expected at such temperature. Furthermore, the stress state will be lower than those given in Figure 1 and more in the power law region ($\dot{\varepsilon} = k\sigma^n$ where $n \simeq 5$) where the effect of subgrain size on creep has been well defined for the case of polycrystalline aluminum(3). Strain-rate-change tests will be planned at all temperatures (900, 1000 and 1100°C) to establish the subgrain size-creep strength relation following the procedure described in the first quarterly progress report(4).

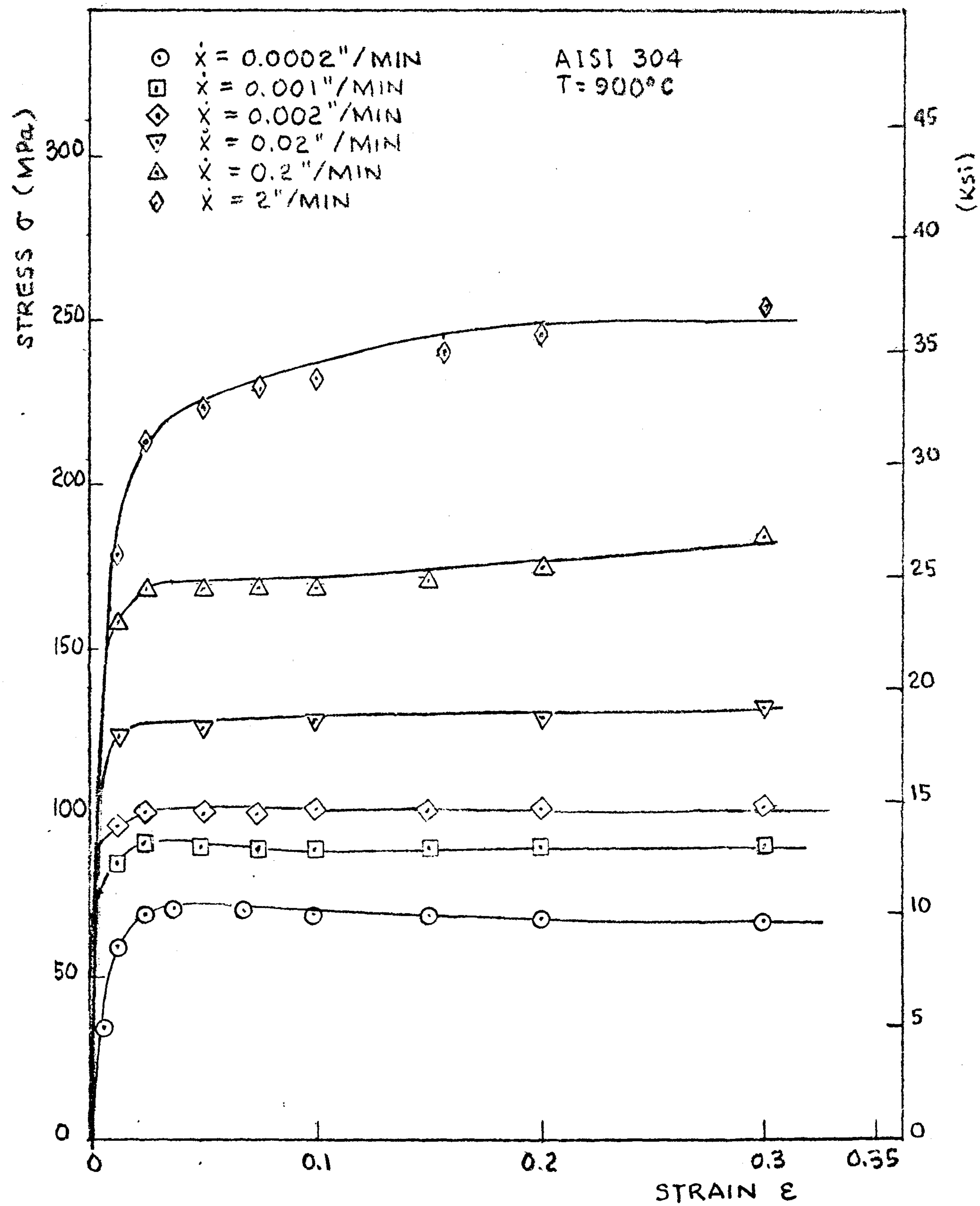

Figure 1. True stress-true strain curves for 304 stainless steel deformed in compression at several strain rates.

E-Brite 26-1

E-Brite 26-1 is an electron beam melted ferritic stainless steel (26 Cr, 1 Mo). Earlier work on this material[2] revealed formation of well developed subgrains during torsional deformation in the temperature range 400 to 1000°C. The subgrain size developed at different flow stresses (plotted as modulus compensated stress) is illustrated in Figure 2. The results are compared with those obtained for 304 stainless steel. At low stresses, $\lambda \propto (\frac{\sigma}{E})^{-1}$ and at high stresses $\lambda \propto (\frac{\sigma}{E})^{-2}$. Such an observation was noted earlier for a number of ferrous alloys[5].

An important aspect of torsion testing is that it permits the attainment of large plastic strains prior to failure. Thus, one has available a simple laboratory tool of studying unusual structures that may be developed at large strains typical of commercial forming processes. By large plastic strain we have in mind strains in the order of $\varepsilon = 15$ (45 twists on our torsion sample); this is typically 30 times the strain that is considered in normal large deformation studies when $\varepsilon \simeq 0.50$ (typical values in compression or tension tests).

We illustrate, in Figure 3, the influence of large strain deformation on the high temperature stress-strain curve for E-Brite 26-1. On the same graph we illustrate the room temperature yield strength of the ferritic stainless steel at room temperature after torsional deformation at high temperature; the yield strength was obtained by compression tests on hollowed-out torsionally deformed samples[4]. The results obtained were quite surprising. The strength at 700°C decreased with continued straining whereas the room temperature yield strength increased with increasing plastic strain. Microscopy studies did not yield a definite explanation for these results. Transmission electron micrographs were prepared and these revealed that subgrains were well developed by a strain of 1.0 with $\lambda = 0.8\mu m$. Further deformation lead to virtually no change in subgrain

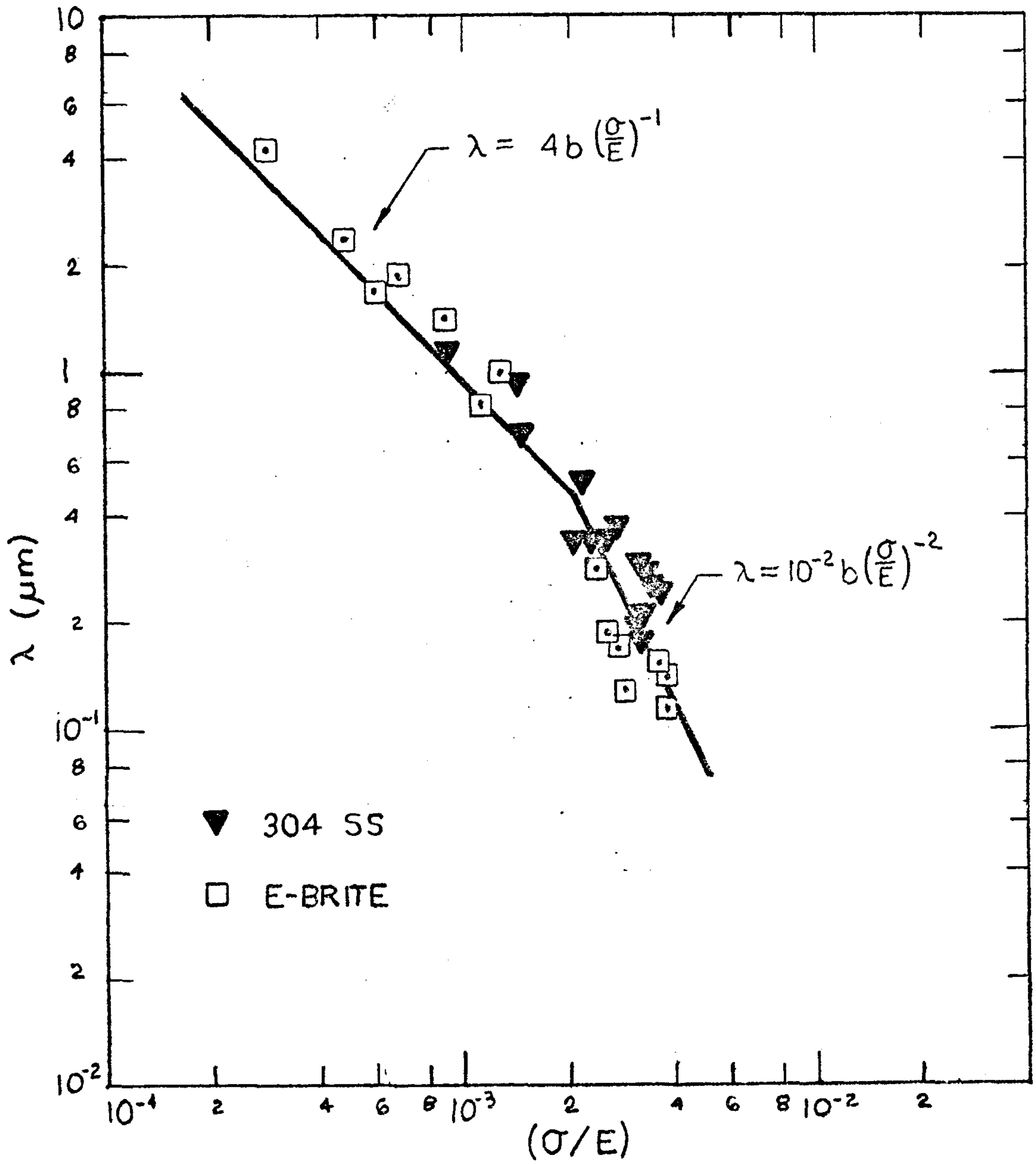

Figure 2. The subgrain size (λ) as a function of the modulus compensated stress (σ/E) for AISI type 304 stainless steel and E-Brite 26-1 (Fe-26 Cr-1 Mo) at low stresses ($\sigma/E < 2 \times 10^{-3}$) $\lambda \propto (\sigma/E)^{-1}$. At high stresses ($\sigma/E > 2 \times 10^{-3}$) $\lambda \propto (\sigma/E)^{-2}$. Subgrains of the same size were formed in the austenitic 304 stainless steel and in the ferritic E-Brite stainless steel at constant σ/E. The above data were taken from Young, Walser and Sherby[2].

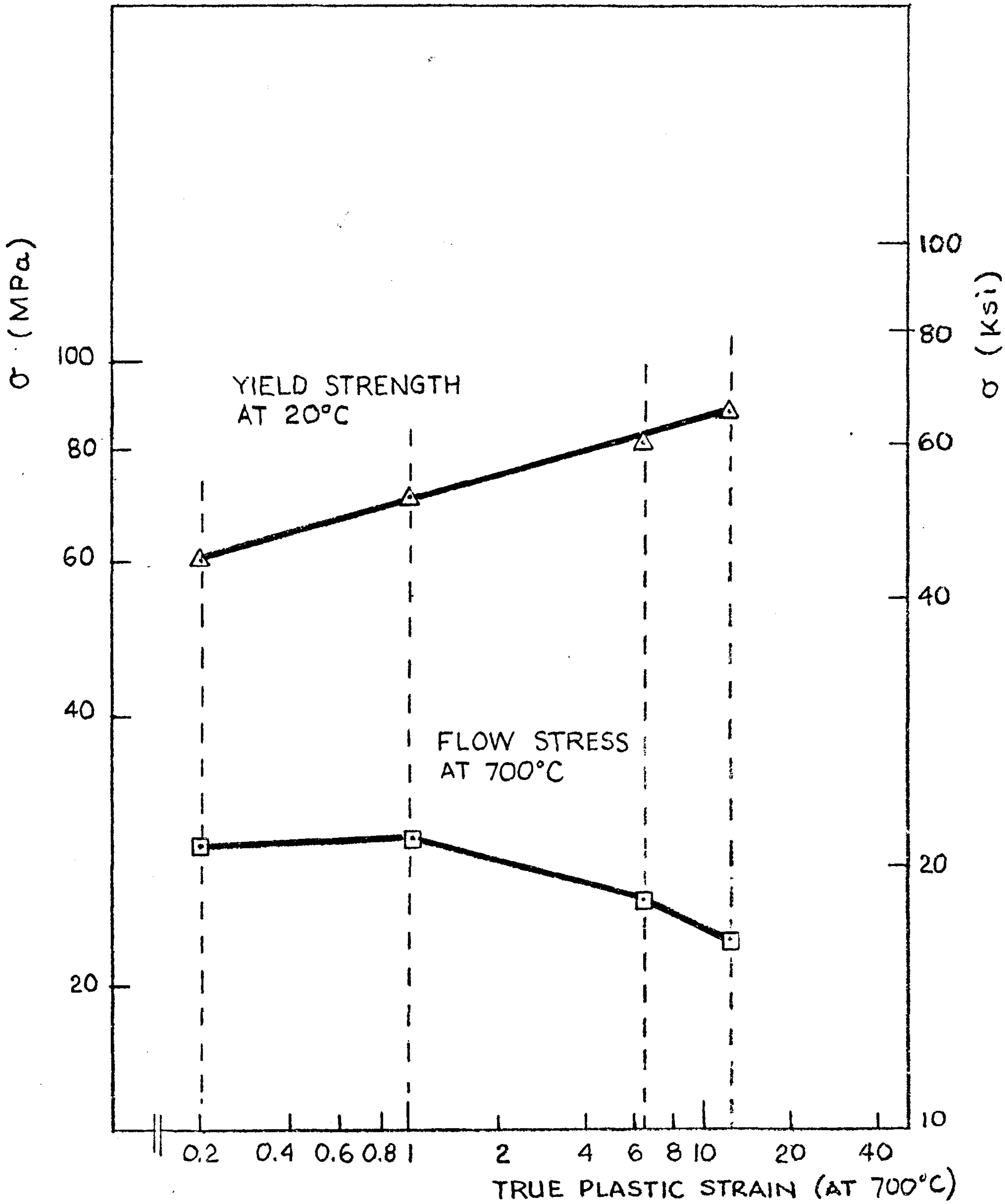

Figure 3. Influence of large strain deformation ($\varepsilon \simeq 15$) at 700°C and $\dot{\varepsilon}$ = .006 S^{-1} on the flow stress at 700°C and on the yield strength at room temperature for E-Brite 26-1.

size with no obvious change in the dislocation distribution within subgrains. One possible explanation for the strength changes observed is that the perfection and nature of the subgrain boundary changes with extensive plastic straining. Thus, one may speculate that as the subgrain boundary becomes more perfect with continued straining, it becomes a less effective barrier at elevated temperature but a more effective one at low temperature. These data thus show the possible importance of taking into account the amount of warm working for optimizing properties at low and at high temperatures. Another possible explanation for the results obtained may center on the development of a texture during large strain deformation. We intend to perform x-ray studies in order to determine the possible factor contributing to the results given in Figure 3.

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4. Rodney Klundt, Bruno Walser and Oleg D. Sherby, First Quarterly Progress Report, September 1, 1974 - December 31, 1974, Contract AT (04-3)-326 PA#38, Department of Materials Science and Engineering, Stanford University, Stanford, California 94305.

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MECHANICAL PROPERTIES OF ULTRA-FINE STRUCTURES DEVELOPED BY WARM WORKING.
SUBGRAIN BOUNDARY STRENGTHENING

(Oleg D. Sherby, Rodney H. Klundt and Bruno Walser)

WARM WORKING. Under ordinary strain rates ($\dot{\varepsilon} \simeq 10^{-4}$ to 1 sec^{-1}) warm working is generally defined as plastic deformation in the range 0.35 to $0.6T_m$ (where T_m is the absolute melting temperature). As the deformation rate is increased the warm working range is shifted to high temperatures. Warm working is characterized (a) by development of fine equiaxed subgrains, (b) by high atom mobility associated with generation of excess vacancies and dislocations during mechanical working and (c) by high ductility of the material. Much of these observations have been developed and quantified by us at Stanford University.

Our current objective is to understand the nature of subgrain boundaries and to assess their importance in enhancing the strength of materials. We are especially interested in learning how to develop stable fine subgrains as a possible means of improving the creep resistance of iron and nickel base alloys.

SUBGRAIN BOUNDARIES. Subgrain boundaries are low angle dislocation boundaries, and have long range stress fields associated with them. The misorientation between boundaries is typically between 0.1 and 2 degrees. The size of subgrains is primarily a function of the applied stress during warm working. Our work (coupled with other investigations) reveal that

$$\lambda \simeq 4b \left(\frac{\sigma}{E}\right)^{-1} \text{ at low stresses, and } \lambda = 10^{-2} \left(\frac{\sigma}{E}\right)^{-2} \text{ at high stresses} \qquad (1)$$

where λ is the subgrain size, b is Burgers' vector ($\approx$ atom spacing), σ is the deformation stress and E is Young's modulus. The perfection of the subgrain boundary appears to be dependent on the strain and temperature of warm working.

STANFORD TORSION MACHINE. Since the size of subgrains decreases as the deformation stress increases, fine subgrains are obtained at high strain rate and/or low temperatures (i.e. at the low end of the warm working range). In order to obtain and study the fine structures developed during high rate warm working it is necessary to quench the sample rapidly in order to "capture" the subgrain structure (before subgrain growth or recrystallization can occur).

The Stanford torsion test machine has been developed to simulate complex forming operations at high strain rates and is ideally suited to perform our "subgrain boundary strengthening" studies. Torsion testing permits attainment of large strain [ε = 5.0 (15 twists for a 1/4" diam. 1" gage length sample) is readily obtained], and strain rates up to 10,000% per second are achievable.

SUBGRAIN BOUNDARY STRENGTHENING. We have found that subgrains increase both the low temperature strength as well as the high temperature strength.

a. Low Temperature. With respect to low temperatures, we have shown, for several materials that

$$\sigma_y = \sigma_o + k\lambda^{-1} \qquad (2)$$

where σ_y is the yield strength at low temperature (in our case 298°K), σ_o is the stress for plastic flow of the material without subgrains and k is a constant related to the strength of the subgrain boundary. We have found that k is a function of the strain and temperature of warm working (Figure 1). It would appear, furthermore, that fine subgrain boundaries can be better obstacles to plastic flow than high angle grain boundaries (Figure 2). An unusual feature of subgrains with respect to mild steels is that their presence appears to inhibit the yield point phenomenon and Luders band propagation (Figure 3).

b. High Temperature. By means of stress-drop tests we have recently uncovered, apparently for the first time, the influence of subgrain size on the creep resistance of simple metal systems at elevated temperature. Our results reveal that

$$\dot{\varepsilon} \propto \lambda^3 \sigma^8 \qquad (3)$$

where $\dot{\varepsilon}$ is the creep rate. Thus, if the subgrain size is decreased by a hundred fold the creep rate is reduced by one million times!! We believe that the primary source of strengthening in dispersion hardened materials is the presence of fine stable subgrains developed during working of such materials. We graphically illustrate our ideas in Figs. 4,5 and 6.

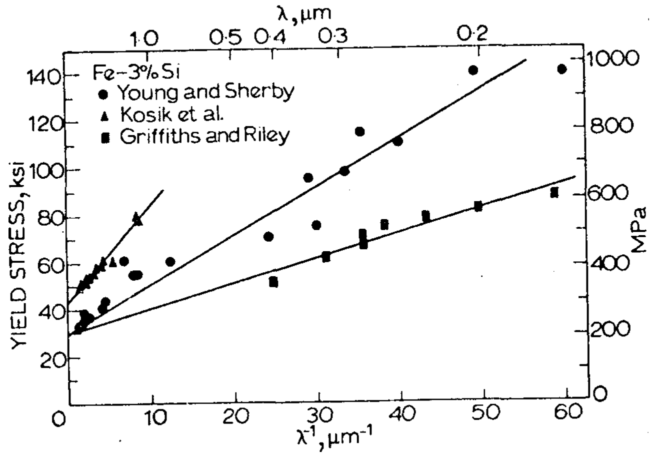


Figure 1. The room-temperature yield strength as a function of λ^{-1} (subgrain size^{-1}) for Fe-3%Si from several investigations: the data suggests that a linear relation is obtained, with reasonable agreement of σ_0 obtained from various investigations. The slope k relates to the strength of the subgrain boundary and appears to be a function of the temperature of prior working.

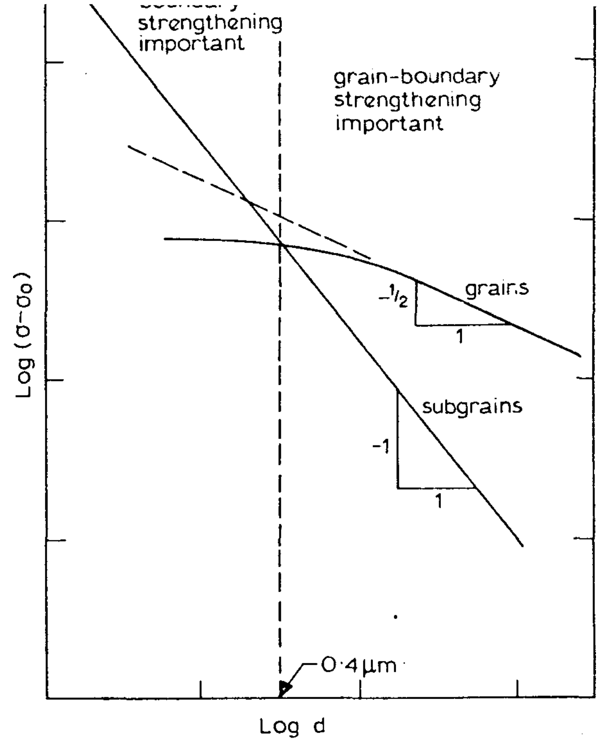


Figure 2. Schematic representation of the trends predicted by grain and subgrain-boundary strengthening: at a grain size less than about 0.4μm, subgrains would apparently be stronger than grains of the same size.

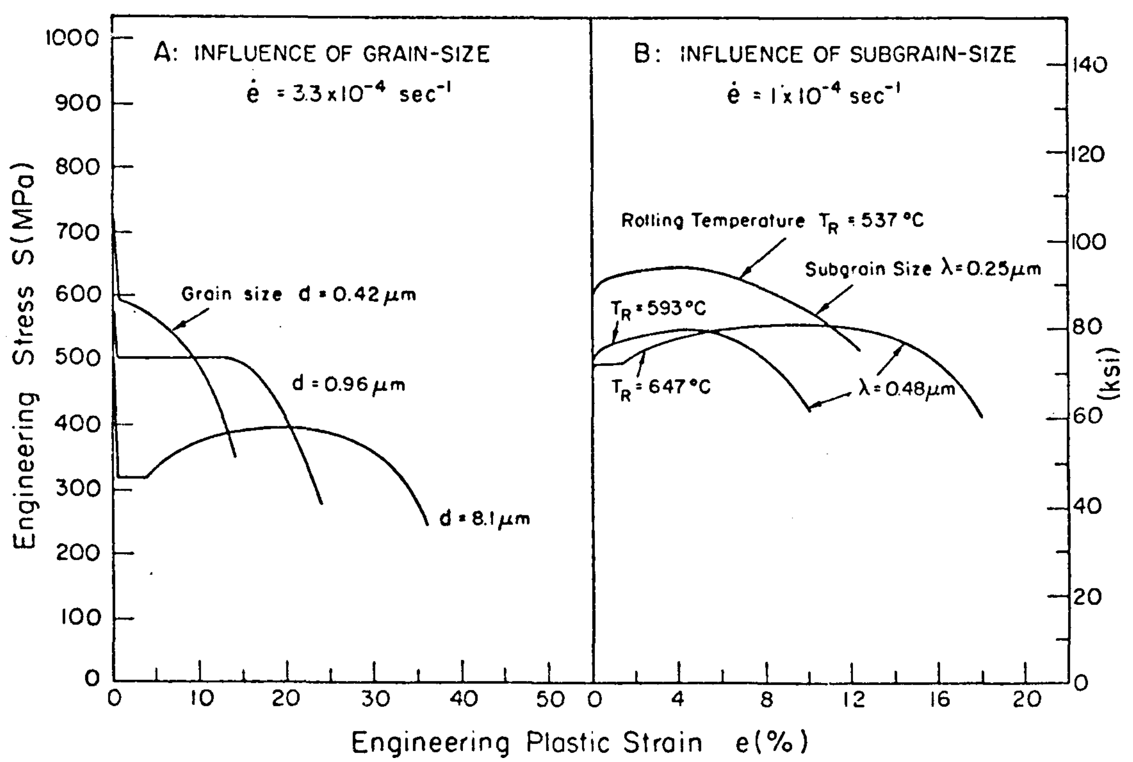
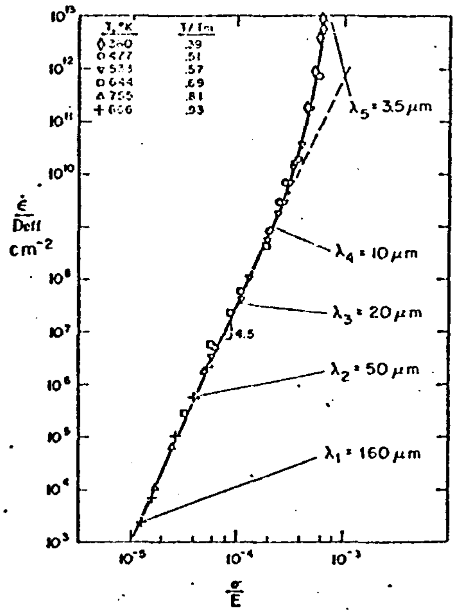
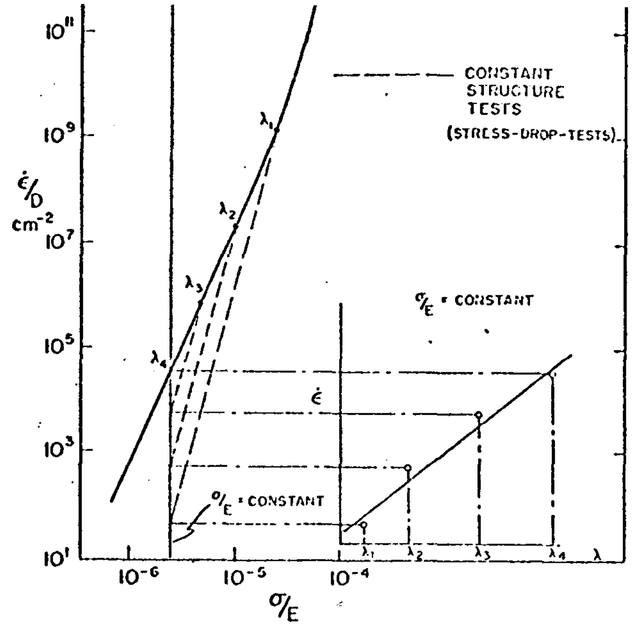


Figure 3. Influence of fine subgrains and grains on the room temperature tensile stress-strain curves for a 1017 carbon steel. The yield phenomenon and accompanying Luders band present in the fine grained material appears to be inhibited by the presence of subgrains. The different subgrain sizes were obtained by warm rolling at different temperatures.





5. The steady state creep rate-stress relation is not unique because the structural state is a variable, i.e. a different subgrain size exists at each stress. In order to determine the influence of subgrain size on the creep rate it is necessary to do constant structure creep tests by "stress-drop" tests.

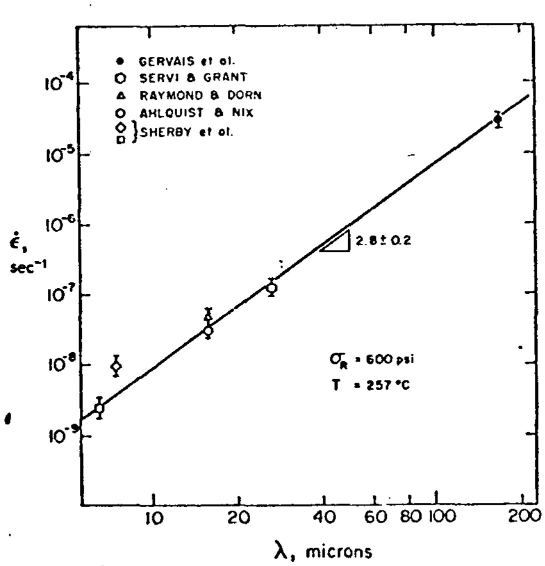
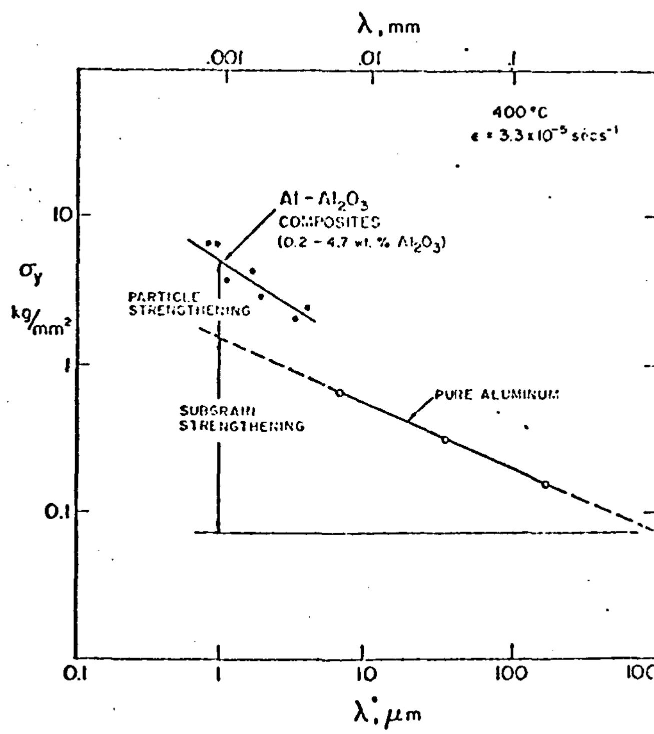


Figure 5. The creep rate dependence upon subgrain size in high purity aluminum as assessed by stress drop tests
A relation $\dot{\varepsilon}|_{\sigma,T} \propto \lambda^3$ is obtained



7. Influence of subgrain size on the yield or flow stress of aluminum and $Al-Al_2O_3$ composites at 400°C and $\dot{\varepsilon}=3 \times 10^{-5}$ per sec. The results obtained suggest that the high strength of the aluminum-alumina composites can be attributed primarily to subgrain strengthening.