Effect of Annealing on Structure and Properties of a Dispersion Strengthened Superalloy, IN-853

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Dispersion strengthened nickel-base superalloy bar has been produced by hot extrusion of mechanically alloyed powders. The fabrication methods produce an ultra-fine grained material which resists grain growth up to a critical temperature around 1506 K (2250°F). Above this temperature, a discontinuous grain growth process leads to the formation of coarse elongated macrograins. The material becomes completely coarse grained in short times. Annealing below this temperature produces only slight grain coarsening, and relieves stored strain energy. Changes in properties are related to the grain growth and strain annealing processes.

IMPROVED superalloys are sought to meet the needs of advanced high temperature technology. A particular requirement is for materials which combine high creep strength at intermediate and elevated temperatures with good high temperature corrosion resistance. A new dispersion strengthened nickel-base superalloy, designated IN-853,* combines the corrosion resistance and intermediate temperature strength of a conventional γ' hardened nickel-base superalloy with the high temperature strength and stability characteristics of a dispersion strengthened metal.[1]

*This alloy is produced commercially by the Huntington Alloy Products Division of the International Nickel Company, Inc., as INCONEL® alloy MA-753.

The alloy is made by extrusion of powder produced by the newly-developed mechanical alloying process. Mechanical alloying consists of processing a blend of raw material powders in a dry, high energy ball mill or attritor in which the charge is driven by a rotating paddle.[1] The combined processes of cold welding and comminution[2] which occur during mechanical alloying lead to the production of composite powder particles containing an intimate dispersion of refractory oxide particles.

Extruded IN-853 bar is an ultra-fine grained material which is strong at ambient temperature. Exceptional elevated temperature tensile and creep rupture strengths are developed after heat treatments designed to produce a coarse elongated grain structure in the material. This paper discusses the effects of annealing on grain growth, and mechanical properties of IN-853.

EXPERIMENTAL PROCEDURES

Material

The IN-853 used had a nominal matrix composition of 19 wt pct chromium, 1.2 wt pct aluminum, 2.4 wt pct titanium, 0.07 wt pct zirconium, 0.007 wt pct boron, balance nickel, dispersion strengthened with yttrium oxide. The Y_2O_3 content was 2.25 vol pct. Excess oxygen (~0.5 wt pct) and nitrogen (~0.12 wt pct) introduced into the powder during the milling process, form secondary dispersoids in combination with part of the matrix aluminum and titanium. Mechanically alloyed powder, produced from the raw materials shown in Table I, was packed in mild steel cans which were sealed by fusion welding, and extruded to bar in a 8.9 cm diameter press. A range of extrusion temperatures and ratios were used in the preparation of bar.

Mechanical Tests

Tensile tests at both room and elevated temperature were performed on samples of bar in the as-extruded condition and after various anneals at temperatures ranging from 1311 K (1900°F) up to 1589 K (2400°F). 1311 K (1900°F) stress rupture tests were performed on samples of bar: a) as-extruded; b) annealed for 10 h at 1450 K (2150°F); or c) heat treated 1/2 h at 1589 K (2400°F) in argon, air cooled, then aged 24 h at 978 K (1300°F).

Metallography

Samples for metallographic study were polished and etched electrolytically in 10 pct sulfuric acid. The fine grain structure was examined using electron microscopy of germanium-shadowed parlodion replicas. Foils for transmission microscopy were thinned by a jet technique. Samples were dished in DISA A2 electrolyte and final polishing was done at room temperature in 10 pct perchloric/acetic at 20 volts and 200 milliamps.

Table I. Raw Materials Used in Mechanical Alloying of In-853

Component	Particle Size
Type 123 Carbonyl Nickel	4-7 μm
Chromium	−200 mesh
Ni/16.5 Al/28 Ti master alloy	−200 mesh
Ni/29 Zr master alloy	−200 mesh
Ni/17 B master alloy	−200 mesh
Y_2O_3	200-300Å

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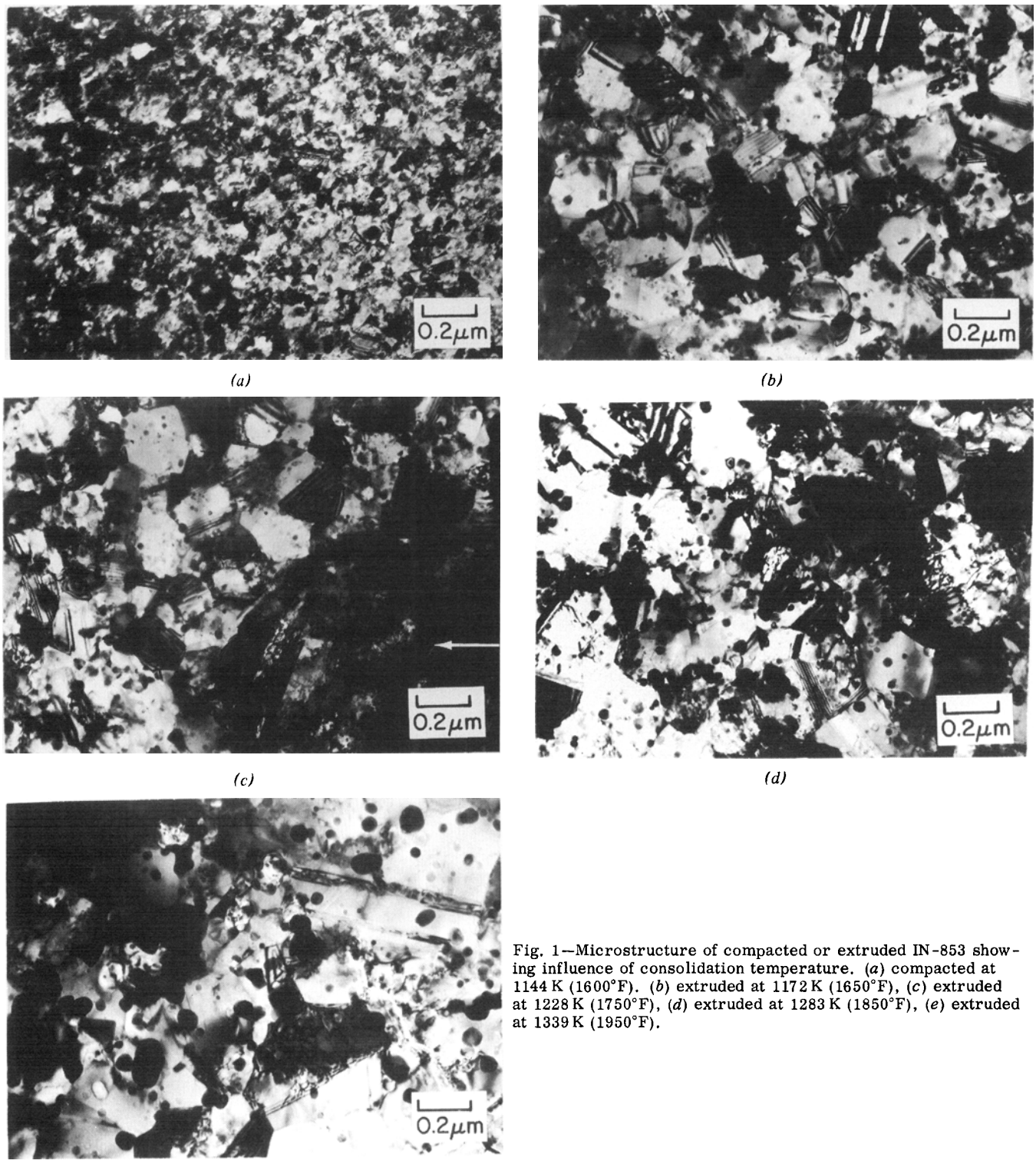


Fig. 1—Microstructure of compacted or extruded IN-853 showing influence of consolidation temperature. (*a*) compacted at 1144 K (1600°F). (*b*) extruded at 1172 K (1650°F), (*c*) extruded at 1228 K (1750°F), (*d*) extruded at 1283 K (1850°F), (*e*) extruded at 1339 K (1950°F).

RESULTS AND DISCUSSION

Effect of Annealing on Grain Structure

Primary Recrystallization: IN-853 bar is in general recrystallized immediately after extrusion to a fine grain size which increases with the extrusion temperature. The grains formed are equiaxed and have boundaries which can be clearly deliniated by etching. Despite this clear evidence that the structure is recrystallized with high angle grain boundaries, it is interesting that the material still has a relatively high dislocation content. By measuring dislocations in a number of grains in bar extruded at 1339 K (1950°F) order of magnitude calculations indicated a total dislocation content $\sim 10^9$ cm/cm^3. The deformation substructure is thus strongly pinned by the dispersoid and resists being swept away during the original recrystallization process. Transmission micrographs in Fig. 1 show the structure of bar extruded or compacted at temperatures ranging between 1144 K (1600°F) and 1339 K

(1950°F). Due to the rather low matrix aluminum level the γ' content of IN-853 in fully aged condition is less than 15 pct, and the γ' solvus temperature is below 1230 K (1750°F). It is unlikely that the presence of γ' has any significant effect on the recovery or recrystallization behavior of this alloy.

Material consolidated at the lowest temperatures shows evidence of partial recrystallization but no dislocation substructure can be resolved in most of the unrecrystallized material. Fig. 1(*a*) shows the structure of powder compacted to full density at 1144 K (1600°F) in an extrusion press with a blank die. Recrystallized grains present in the material had an average size considerably less than 0.1 μm.

Recrystallization is not quite complete after extrusion at temperatures at least up to 1228 K (1750°F). The bar retains small regions of unrecrystallized material which have a very high dislocation density residual from the intense deformation of the mechanical alloying process. An example of such a region is shown in Fig. 1(*c*) which is a foil from a bar extruded at 1228 K (1750°F).

Recrystallization is complete after extrusion at higher temperatures and the grain size increases progressively with extrusion temperature although remaining very fine. The sequence of photographs in Fig. 1 show this effect. Lineal analysis of transmission and replica micrographs was used to determine the average grain size. Material extruded at 1172 K (1650°F) (Fig. 1(*b*)) had an average grain intercept length (N_L) about 0.12 μm; in material extruded at 1339 K (1950°F) (Fig. 1(*e*)) N_L had a value about 0.23 μm; in material extruded at 1478 K (2200°F) N_L was increased to about 0.6 μm. The average grain size as indicated by N_L was related to the extrusion temperature (T) by the relation

$$N_L \propto \frac{1}{T^2}$$

shown in Fig. 2.

Annealing: has different effects on the structure and properties of IN-853 depending on the temperature. The fine grained structure of extruded bar resists growth to temperatures around 1311 K (1900°F). Webster[3] and Inman, *et al*,[4] have observed stability of structure in dispersion strengthened materials, while inhibition of normal grain growth by dispersed particles has long been recognized as a possible mechanism to promote secondary recrystallization in fine grained silicon steel.[5] Annealing between 1311 K (1900°F) and about 1506 K (2250°F) causes slight grain growth, which is both time and temperature dependent, as well as growth of carbide particles throughout the material. The transmission electron micrographs in Fig. 3 which were chosen to be fairly typical examples, show the general effect of annealing on material extruded at 1339 K (1950°F). During the first stages of the annealing process the dislocations tend to become concentrated near grain boundaries (Fig. 3(*a*)). Numerous concave grain boundaries indicate that growth of grains with low dislocation density is occurring at expense of heavily dislocated material and annealing twins are common within these growing grains. At longer annealing times the substructure is gradually resolved until the dislocation content of most of the material is low (Fig. 3(*b*)). Occasional pileups are

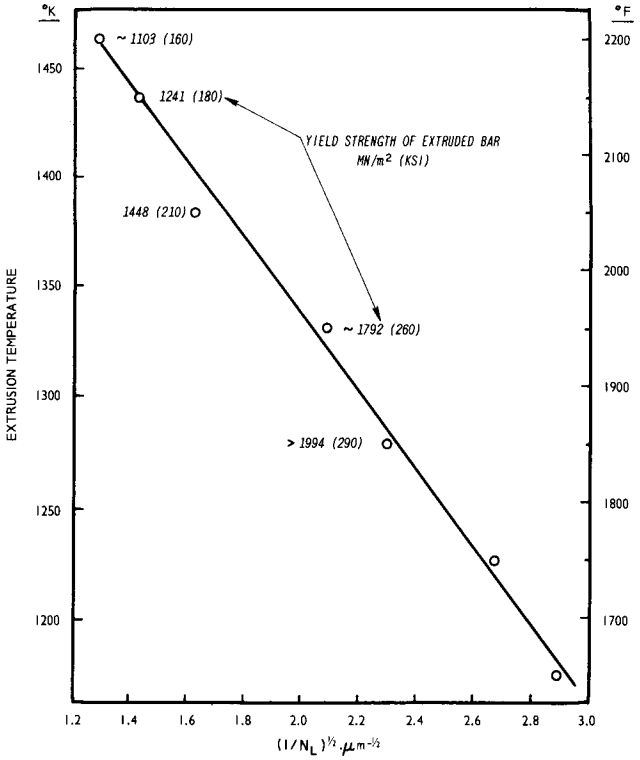


Fig. 2—Relation between extrusion temperature and grain size of extruded IN-853 bar.

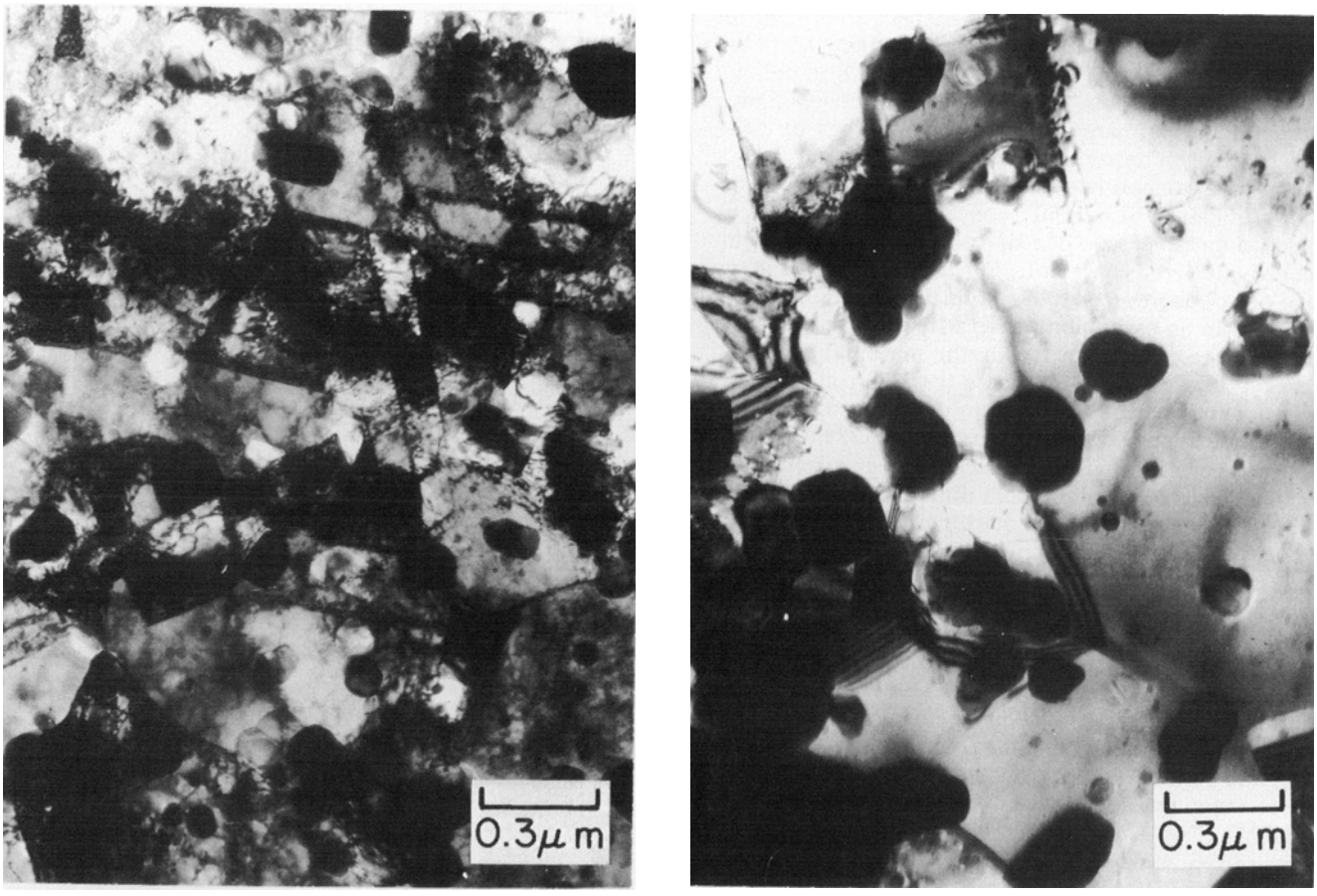
observed between particles and some dislocation arrays observed within grain boundaries.

Secondary Grain Coarsening: At a critical temperature near 1533 K (2300°F) rapid grain growth takes place, resulting in a structure generally containing coarse elongated grains (Fig. 4(*a*)). After this treatment much of the material has a very low dislocation content. In some regions dislocations are retained as tangles which seem organized in an irregular subcell structure such as shown in Fig. 4(*b*). In this condition IN-853 develops exceptional high temperature strength and stress rupture behavior. Although the mechanism of this discontinuous grain growth process is not known, it resembles secondary recrystallization behavior observed in other dispersion strengthened materials,[6] and the grain growth behavior of doped tungsten.[7]

As can be seen from the preceding discussion, IN-853 has a fairly high dislocation density in the fine grained as-extruded condition. The dislocation substructure is not organized to a large extent within grain or subcell boundary surfaces. Recovery of dislocations to grain boundaries within fine grained material during subcritical annealing eliminates the grain coarseneing response. Immobilization of the fine grain boundaries, removal of nucleation sites, and reduction of driving force for the grain coarsening through reduction of the dislocation density, may all be factors contributing to this effect.

Effect of Annealing on Mechanical Properties

Room Temperature Properties: In the as-extruded condition, IN-853 bar has high room temperature tensile strength due to the extremely fine grain size of the extruded product and residual strain energy de-



(a) (b)

Fig. 3—Structure of IN-853 after different annealing treatments. (*a*) annealed 2 h at 1450 K (2150°F), (*b*) annealed 48 h at 1450 K (2150°F).

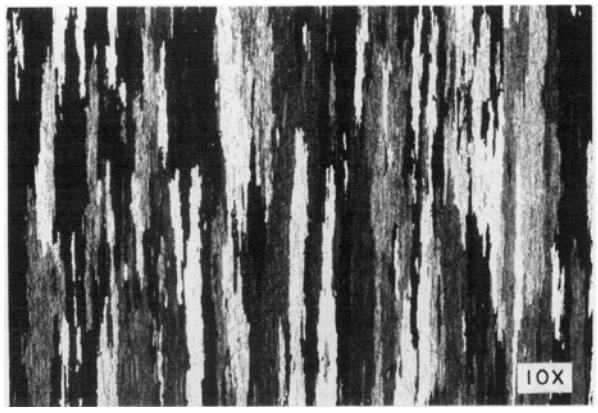
rived from the mechanically alloyed powders. The variation of yield strength with the extrusion temperature and the grain size is indicated on Fig. 2. The effect of annealing on room temperature mechanical properties is shown in Fig. 5. For these tests bar which was extruded at 1339 K (1950°F) was annealed for 10 h at different temperatures over the range 1255 K (1800°F) to 1589 K (2400°F) (Table II). The yield and tensile strengths decrease progressively with annealing temperature above 1311 K (1900°F) to about 1533 K (2300°F) where grain coarsening occurs. Recovery at temperatures above 1422 K (2100°F) causes a drop in the yield/tensile ratio and increased work hardening capability. The material furthermore shows a marked yield point and yield point extension when annealed over this temperature range. Grain growth caused by the heat treatments was determined by lineal analysis on replica micrographs. The relation between yield stress and average grain intercept length (N_L) drawn as a Petch type plot in Fig. 6 shows that the change in yield strength is related to the observed increase in grain size.

The yield stress is also influenced by the degree of recovery and particle growth which may occur. Clearly interactions between the dispersoid particles and dislocations should play a role in determining strength in this material. The relation in Fig. 6 takes no account of the interparticle spacing and size or of dislocation subcell structure. While the value of $K = 3.16 \cdot kg^{-3/2}$ is comparable with values reported for very fine grained dispersion strengthened aluminum,[8] it is higher than the values ($0.5 \cdot kg \cdot mm^{-3/2}/2.0 \cdot kg \cdot mm^{3/2}$) generally reported for nickel.[9] The extrapolated curve also gave a value of $\sigma^0 \doteq -344.7$ MN/m^2. Fig. 7 illustrates schematically how greater recovery in material annealed to coarser grain sizes at higher temperatures could lead to high value for K and a negative intercept for the curve. Annealing at temperature T_1, giving rise to the annealed grain size d_1, produces a greater drop

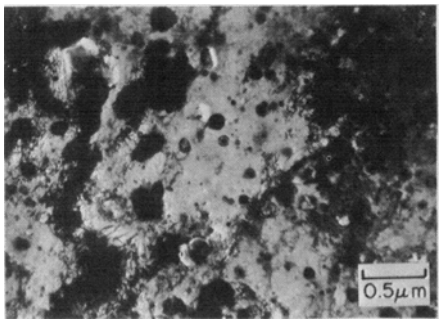
Table II. Room Temperature Tensile Properties of Annealed In-853

Heat Treatment Hours at Temperature, °K	0.2 pct Yield Strength MN/m^2, ksi	U.T.S. MN/m^2, ksi	Elong, pct	R.A., pct
10/1255	1606 (233)	1641 (238)	0.5	1.5
10/1367	1475 (214)	1475 (214)	2	8
10/1422	1227 (178)	1289 (187)	4	5.5
10/1450	1062 (154)	1241 (180)	5	6.5
10/1478	979 (142)	1207 (175)	6	8.5
10/1506	738 (107)	1131 (164)	11	14.5
10/1533	689 (100)	1007 (146)	12	16.5
2/1589	696 (101)	1027 (149)	12	15.5
10/1367 + 2/1589	641 (93)	986 (143)	11	16
10/1422 + 2/1589	869 (126)	1161 (167)	8	11.5
10/1450 + 2/1589	848 (123)	1179 (171)	10	14.5
10/1478 + 2/1589	848 (123)	1207 (175)	12	15
10/1506 + 2/1589	696 (101)	1145 (166)	15	13
10/1533 + 2/1589	669 (97)	1013 (147)	12	13

in yield strength due to recovery than does annealing at the lower temperature T_2 which produces a smaller grain size d_2. As a result the plot of yield stress against grain size lies below and has a steeper slope than would be expected if no recovery took place. The plot is a straight line as shown in Fig. 6 only when the drop in yield strength due to recovery is also proportional to the change in grain size.



(a)



(b)

Fig. 4—Structure of IN-853 after grain coarsening.

Elevated Temperature Properties: Elevated temperature tensile and stress rupture properties of IN-853 differ according to the material state and heat treatment. Data for tests at 1255 K (1800°F), 1311 K (1900°F) and 1367 K (2000°F) on material in a) as-extruded, b) fine grained annealed, and c) grain coarsened conditions (Table III) show the high strength developed through grain coarsening. The alloy shows low strength in both the as-extruded and annealed conditions, but a marked difference in deformation characteristics is observed (Fig. 8). In contrast with the behavior of as-extruded material, the alloy did not neck in the tensile gage section after annealing, although similar elongations were obtained.

To examine this difference in more detail, elevated temperature tensile tests were made at various strain rates on IN-853 bar. Material was in the as-extruded condition or given one of several different recovery heat treatments to develop a range of room temperature yield strengths down to that of secondary recrystallized material. Tests were performed at temperatures ranging from 922 K (1200°F) up to 1367 K (2000°F), and at strain rates ranging from 0.001/min. up to 1.0/min. Strain rate exponents ranged from 0.15 at 922 K (1200°F) down to 0.11 at 1367 K (2000°F) for extruded material; and between 0.1 at 922 K (1200°F) down to 0.03 at temperatures above 1200 K (1700°F) for coarse grained recrystallized material. Strain rate exponents were generally close to 0.15 for fine grained recovered IN-853 over the whole temperature range. The grain size of annealed specimens varied by a factor of at least three but the elevated temperature flow stress varied little with the recovery annealing treatment despite considerable variations in room temperature

Table III. Elevated Temperature Mechanical Test Data on In-853 Bar in Extruded, Fine Grain Annealed and Coarse Grain Annealed Conditions

Material Condition	Test Temp, °K	0.2 pct Yield Stress at Strain Rate 0.005/min MN/m²	(ksi)	U.T.S. at Strain Rate 0.05/min MN/m²	(ksi)	Elong, pct	R.A., pct
		Tensile Tests					
As Extruded	1255	35.9	(5.2)	54.0	(7.8)	56	64
Annealed 10/1450K	1255	29.0	(4.2)	62.0	(9.0)	38	
Annealed 2/1589K	1255	159.0	(23.0)	179.0	(25.9)	15	29
As Extruded	1311	19.3	(2.8)	32.4	(4.7)	60	64
Annealed 10%1450K	1311	13.8	(2.0)	29.6	(4.3)	49	
Annealed 2/1589K	1311	141.0	(20.4)	159.0	(23.1)	12	27
As Extruded	1367	10.3	(1.5)	20.7	(3.0)	63	64
Annealed 10/1450K	1367	9.7	(1.4)	20.7	(3.0)	39	
Annealed 2/1589K	1367	129.0	(18.7)	147.0	(21.4)	12	25

Material Condition	Stress MN/m²	(ksi)	Rupture Life, h	Elong, pct	R.A., pct
	1900°F Stress Rupture Tests				
As Extruded	13.8	(2.0)	8.4	16	9
	20.7	(3.0)	0.6	36	22
Annealed 10/1450K	10.3	(1.5)	14.6	12	6
	17.2	(2.5)	0.9	20	14
	27.6	(4.0)	0.1	30	22
Annealed 2/1589K*	121.0	(17.5)	100.0	8	8

*Typical value for extruded bar fully heat treated 2/1589 + 2/978K.
†Strain rate increased ten-fold after 0.2 pct offset.

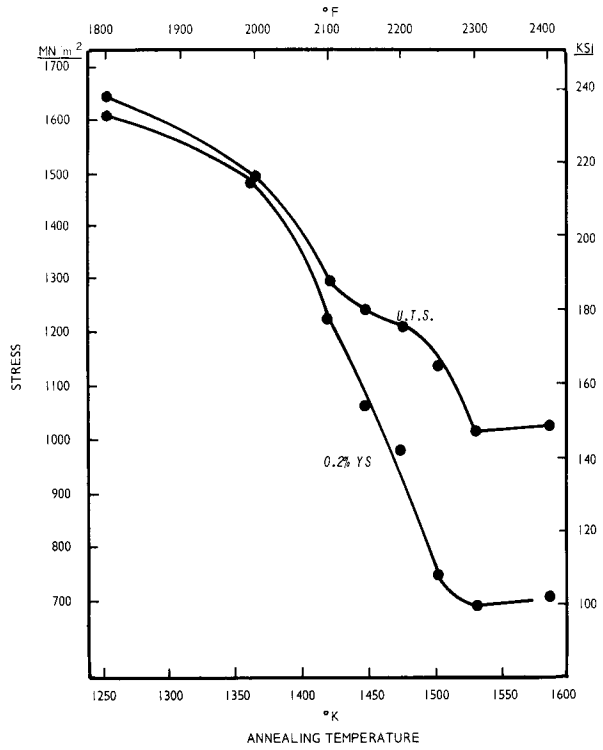


Fig. 5—Room temperature strength of IN-853 after annealing for 10 h at various temperatures.

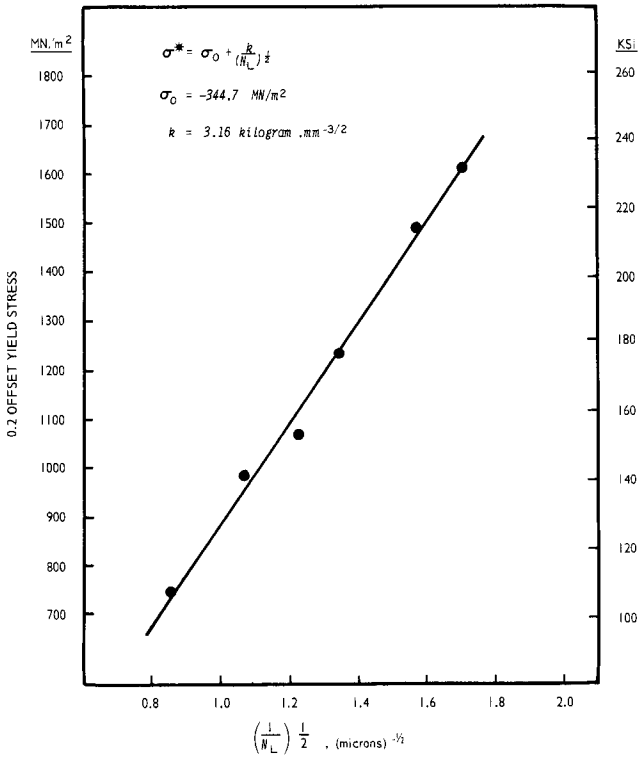


Fig. 6—The relation between yield stress and average grain intercept length (N_L) after annealing heat treatments.

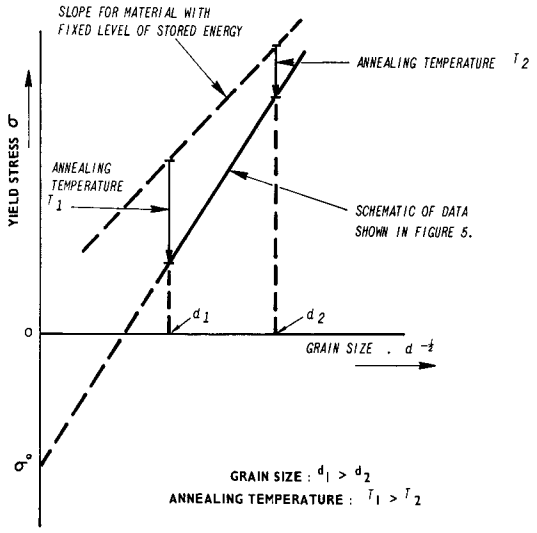


Fig. 7—Illustrating how recovery of stored energy may influence behavior of yield strength as a function of grain size.

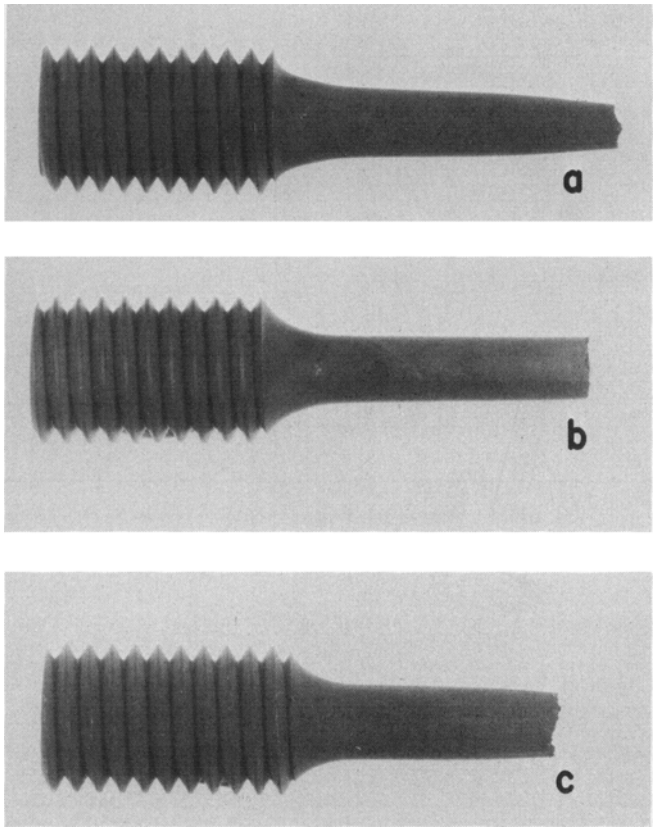


Fig. 8—Tensile deformation of IN-853 tested at 1311 K (1900°F) in different heat treated conditions. (*a*) as-extruded, (*b*) annealed 10 h/1450 K (2150°F), (*c*) annealed 2 h/1589 K (2400°F).

strength (Fig. 9). Coarse grained material was much stronger and showed a different variation of strength with temperature.

Activation energies for the high temperature deformation processes were estimated from the test data. This was done by plotting the true strain rate corresponding to a fixed true flow stress versus reciprocal temperature and determining the slope of the graph. Results are summarized in Table IV. At the high temperatures (1144 K to 1367 K) (1600°F to 2000°F) this activation energy varied with the degree of recovery as illustrated by a plot of activation energy versus room temperature yield strength (Fig. 10); it ranged from about ~712 kilojoules/mole for extruded material, down to ~325 kilojoules/mole for material annealed for 48 h at 1450 K (2150°F). Coarse grained

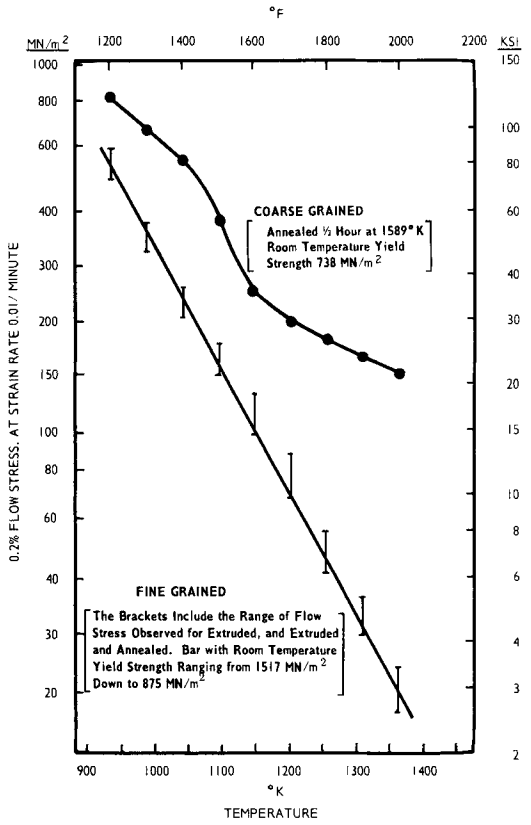


Fig. 9—Temperature variation of flow stress at 0.01/min strain rate for extruded and heat treated IN-853 bar.

Table IV. Activation Energies for High Temperature Deformation of Annealed In-853

Annealing Treatment	Room Temperature Yield Strength MN/m²	(ksi)	Activation Energy Stress MN/m²	(ksi)	Temperature Range, °K	Q Kilojoules/mole
As extruded	1517	(~220)	138	(20)	1144-1367	720
			138	(20)	922-1033	~260
2 h/1450K	1275	(185)	138	(20)	1144-1367	~527
			138	(20)	922-1089	331
10 h/1450K	1089	(158)	138	(20)	922-1367	427
48 h/1450K	896	(130)	138	(20)	1200-1367	347
			138	(20)	922-1144	414
0.5 h/1589K	738	(107)	172	(25)	1200-1367	1000
			552	(80)	922-1033	297

recrystallized material also showed high activation energies for high strain rate deformation in this temperature range. Similar high values of apparent activation energy have been reported for creep in coarse grained material[10,11] though these values cannot be explained at the present time.

At lower temperatures (922 K to 1089 K) (1200°F to 1500°F) activation energies ranging between about 251 kilojoules/mole and 377 kilojoules/mole were observed for all of the different heat treated materials.

The results show that the mechanism controlling deformation in IN-853 varies with the degree of recovery and the test temperature. At low temperatures deformation appears to be governed by mechanisms having activation energies typical of self diffusion which suggests control by dislocation motion. At higher temperatures the same holds true for material which

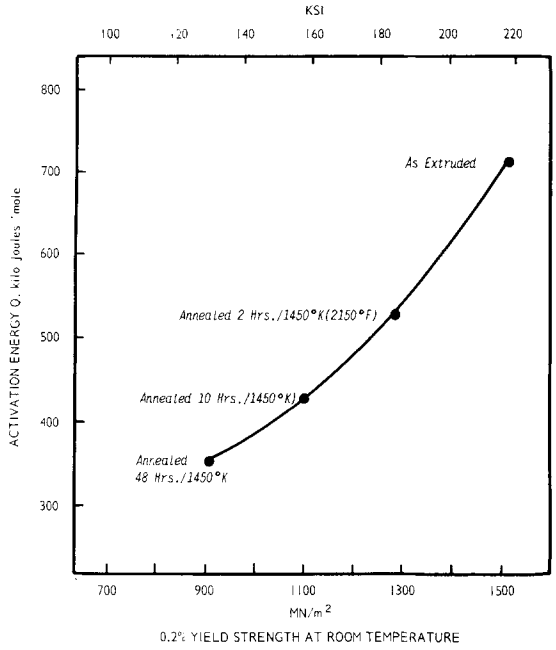


Fig. 10—Relation between activation energy for deformation between 1144 K (1600°F) and 1367 K (2000°F) and degree of recovery as indicated by room temperature yield strength.

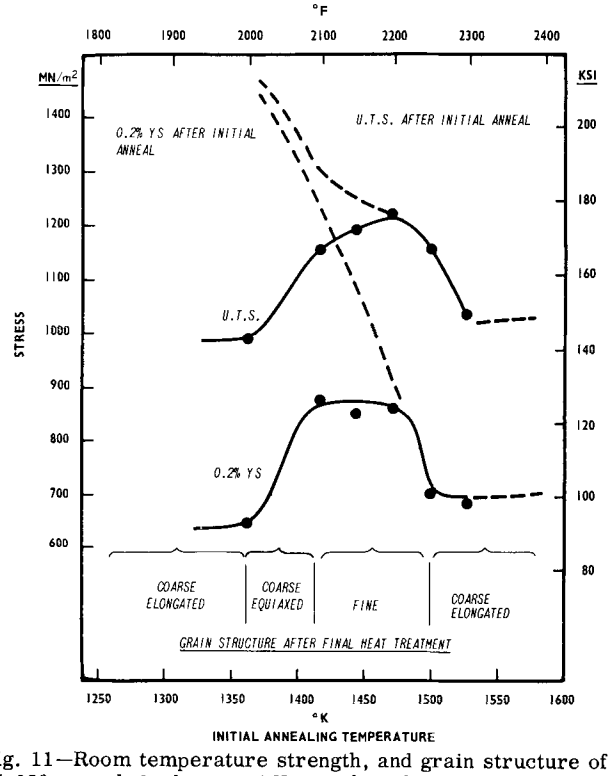


Fig. 11—Room temperature strength, and grain structure of IN-853 annealed 2 h at 1589 K (2400°F) after annealing for 10 h at various temperatures.

has been given heat treatments to produce a substantial degree of recovery.

Grain Coarsening and Mechanical Properties

Grain coarsening in IN-853 is influenced by prior heat treatment. Annealing below the critical temperature can modify the subsequent grain growth response or eliminate it entirely. Data from Table II plotted in

Fig. 11 shows the effect on room temperature strength and grain structure of annealing for 2 h at 1589 K (2400°F) samples which had been previously annealed for 10 h at various temperatures. Grain coarsening to elongated grains occurred in samples given prior anneals up to 1367 K (2000°F) and tensile properties were similar to material given a single anneal above the critical temperature to achieve coarsening. As the prior annealing temperature was increased from about 1367 K (2000°F) to 1423 K (2100°F) the material coarsened to progressively more equiaxed structures and the coarsening response gradually degenerated. Prior annealing in the temperature range 1423 K to 1506 K (2100°F to 2250°F) destroyed the grain coarsening behavior and the material remained fine grained after annealing at 1589 K (2400°F). Some further growth of the fine grains did occur on annealing at 1589 K (2400°F), which caused a further reduction in strength for samples annealed initially at temperatures up to 1478 K (2200°F). Annealing at 1589 K (2400°F) had little effect on the properties of samples annealed initially at temperatures between 1578 K (2200°F) and 1533 K (2300°F) because these were already coarsened to elongated grains.

CONCLUSIONS

1. In the extruded condition, dispersion strengthened nickel-base superalloy bar, made from mechanically alloyed powder, generally has a recrystallized fine grained structure of less than one micron diameter.
2. Annealing above a certain critical temperature, ~1506 K (~2250°F), causes an abrupt increase in grain size to a coarse elongated grain structure (typically 5 mm. × 0.2 mm.).
3. Annealing between 1367 K (2000°F) and 1506 K (2250°F) causes recovery and slight grain growth, and prevents the rapid grain growth behavior.
4. As-extruded bar is strong at room temperature and weak at high temperature. Material heat treated to a coarse elongated grain structure has lower room temperature tensile properties but high elevated temperature strength.
5. High activation energies are obtained for high temperature deformation in extruded bar. The mechanisms leading to these values are not understood. Activation energies obtained for high temperature deformation in recovered material suggest that self diffusion is rate controlling in this condition.

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