

Ultrafine-Grained Microstructures and Mechanical Properties of Alloy Steels

R. L. MILLER

Ultrafine-grained microstructures can be developed in a variety of alloy steels by cold-working followed by annealing in the $\alpha + \gamma$ region. Because the annealing temperatures are relatively low and the recrystallized structure is two-phase, grain growth is restricted. Specimens with grain sizes in the range 0.3 to 1.1 μm (ASTM 20 to 16) were obtained in manganese and nickel steels by annealing 1 to 400 hr at temperatures between 450° and 650°C (840° to 1200°F). The expected improvement in yield strength through grain refinement was observed in almost all alloys. Other tensile properties depend on factors such as grain size, austenite stability, and specimen geometry, that determine which of three types of plastic behavior will occur. Transformation of austenite during straining improves the mechanical properties of ultrafine-grained specimens.

THE influence of grain size on the strength of steel has been known for at least a half-century. The quantitative relationship is usually expressed by the Hall-Petch equation

$$\sigma = \sigma_0 + k d^{-1/2} \quad [1]$$

which states that σ , the yield stress, increases as d , the average grain diameter, decreases. (σ_0 and k are experimental constants.) Eq. [1] has been interpreted in terms of various theoretical models and the relation has been verified numerous times by experiment. Armstrong¹ has reviewed much of this previous work. Until recently, this equation as it applies to steel has been limited to a narrow grain size range for the obvious reason that it is difficult to produce a wide range of grain sizes in steel without changing other factors that affect the strength. Morrison² was able to develop ultrafine grains ($d < 10 \mu\text{m}$) in low-carbon, ferritic steel and the lower yield strengths obeyed Eq. [1] for grain sizes as small as 1.6 μm (ASTM No. 15). By refining the grain size in one steel from 30 to 1.6 μm (ASTM 7 to 15), the strength increased from 28,000 to 77,000 psi (193 to 530 MN/m²). Grange³ showed that even in hardened steel a strengthening effect is evident through refinement of prior austenite grains. He developed a rapid heat-treating process to produce austenite grains as small as 2.5 μm (ASTM No. 14) and again the grain size-strength relation predicted by Eq. [1] was found.

Grain size is known to influence other mechanical properties in addition to strength. Morrison and Miller⁴ reviewed the effect of grain size on tensile ductility; effects on other mechanical properties were reviewed by Armstrong.^{1,5} With the possible exception of uniform elongation and high-temperature strength, most other properties are generally improved, or at worst, not affected by grain refinement. By refining the grain size of high-alloy steel plate, Porter and Dabkowski⁶ obtained a considerably lower ductile-to-brittle transition temperature, in addition to a substantial increase in yield strength.

Grain boundaries are believed to act as major barriers to the propagation of slip and to twinning processes which operate within the grains. The opportunity of increasing the strength and improving the notch sensitivity by reducing the distance between these barriers becomes a strong incentive for producing finer grains in steel. Achieving a stable, ultrafine-grained structure is difficult because of the strong tendency for grain growth. Therefore, many fine-grained microstructures are inherently unstable and unless some means is provided for restricting the movement of grain boundaries, they will not remain fine grained. It was found that a stable, ultrafine-grained microstructure can be obtained by annealing cold-worked ferrous alloys at a relatively low temperature in the two-phase, ferrite-austenite region. The separate phases form as extremely small crystals and their mutual impingement restricts grain growth. By this method, grain refinement in steel can be extended beyond what has previously been achieved.

Suitable alloy compositions that respond to this grain refining treatment can be selected from systems which have a multiphase region extending to relatively low temperatures. The addition of either nickel or manganese to iron produces this feature; a two-phase region extends from 910°C (1670°F) to below room temperature. As an example, the generally accepted Fe-Ni phase diagram⁷ is shown in Fig. 1. The equilibrium phases in this region are ferrite (α) of low nickel content and austenite (γ) of high nickel content. In addition to nickel and manganese, other elements that stabilize austenite, such as carbon and nitrogen, are also beneficial. In some cases, additional phases such as carbides may also form, however, the principal phases are $\alpha + \gamma$ and the steels will be referred to as two-phase.

Since appreciable diffusion is required for the formation of these phases, they do not form when alloys are merely heated or cooled at normal rates through the two-phase region. However, it was found that when the stored energy of the steel is increased by cold-working, profuse nucleation of austenite and rapid partitioning of the alloying elements occur during subsequent annealing in the two-phase region. A similar effect is obtained by working the steel at temperatures within the two-phase region. The result is a recrystallized, two-phase microstructure with grains as small as 0.3 μm (ASTM

R. L. MILLER is Scientist, U. S. Steel Corp. Research Laboratory, Monroeville, Pa.

Manuscript submitted November 4, 1971.

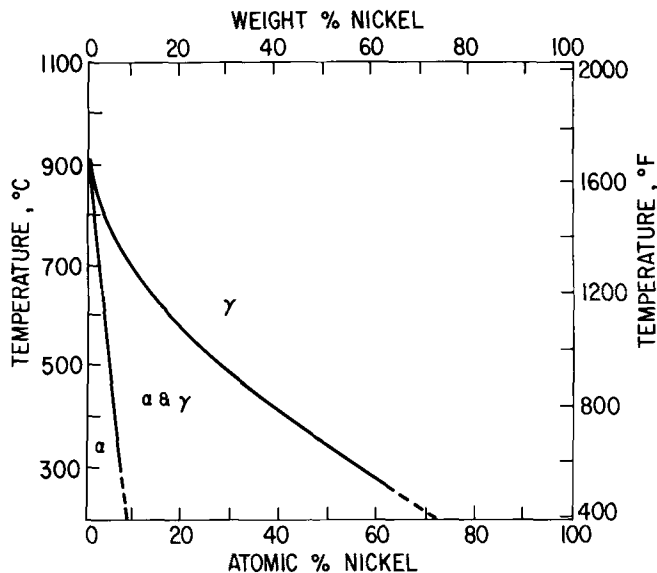


Fig. 1—Equilibrium diagram of the Fe-Ni system below 1100°C.

No. 20). The relation between microstructure and mechanical properties was studied by electron transmission microscopy, light microscopy, X-ray diffraction, hardness, and tensile tests.

MATERIALS AND EXPERIMENTAL PROCEDURE

Table I lists the identification code and the compositions of the steels investigated. Steels B through E were prepared by vacuum melting Grade A104 "Plastiron" and electrolytic manganese, electrolytic nickel, and graphite. Three-in. diam ingots were cast, conventionally hot-rolled, then air cooled. The 9 pct Ni steel, A, was a commercial product in the form of 1 in. plate. All of the steels are martensitic after fully austenitizing and cooling to room temperature.

Cold-working was performed in multiple passes either by rolling or by swaging at room temperature. Generally, the reduction was in the range 60 to 85 pct. Steels that were difficult to work at room temperature were first tempered, then cold-worked and heated in the $\alpha + \gamma$ region. An alternate procedure was warm-working in the $\alpha + \gamma$ region; these specimens were heated in a lead bath at the desired temperature and returned to the bath between each deformation step. The reduction was 75 pct.

Retained austenite measurements were made at room temperature by a standard X-ray technique and a specimen holder designed⁸ to correct for texture present as a result of mechanical working. Lattice parameter measurements were made at room temperature by X-ray diffraction (λ -Cr) from (211) α and (220) γ . Specimens for electron transmission microscopy were thinned by a standard technique⁹ and examined with a Phillips EM-300 microscope. Grain sizes were measured by counting the number of boundaries intersecting a circle superimposed on electron micrographs.¹⁰

Flat tensile specimens with a gage section 1.0 by 0.250 by 0.100 in. and cylindrical specimens with a gage section 1.0 by 0.125 in. diam were strained at room temperature at a crosshead speed of either 0.02

Table I. Composition of Steels, Wt Pct

Identification	Ni	Mn	C	Si	S	N	P	Cr	Mo
A	8.74	0.45	0.12	0.26	0.012	0.009	0.006	0.10	0.025
B	—	5.7	0.11	—	—	—	—	—	—
C	16.3	—	0.053	—	—	—	—	—	—
D	15.1	—	0.009	—	—	—	—	—	—
E	21.0	—	0.053	—	—	—	—	—	—

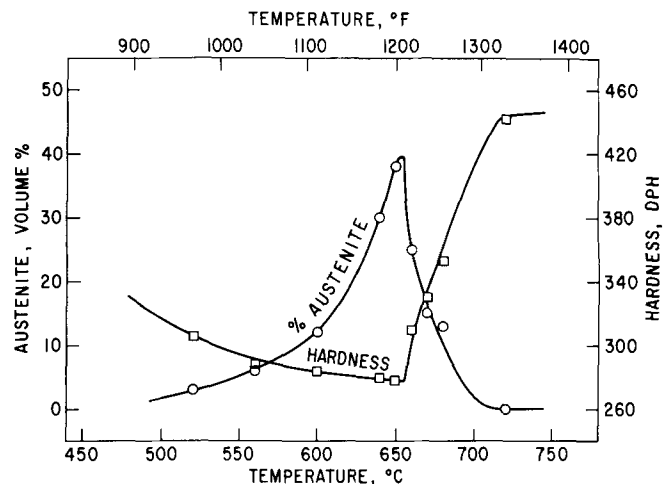


Fig. 2—Hardness and austenite content after 1 h at temperature (Steel B).

or 0.03 in./min. In most cases, two specimens in each condition were tested.

RESULTS AND DISCUSSION

Determining A_s and A_f Temperatures

Since the extent of the ferrite-plus-austenite region varies with the composition of the steel, tests were conducted to determine the temperature at which austenite started to form (A_s) and the temperature at which the steel became fully austenite (A_f). In carbon and low-alloy steels, A_s can be determined from hardness measurements by assuming that in a series of specimens heated to selected temperatures, an increase in hardness at room temperature indicates that austenite was present at the elevated temperature. However, in high-alloy steels, a considerable amount of austenite can form before any hardness increase is observed. In addition, the austenite that forms usually cannot be detected by light microscopy; therefore, X-ray diffraction was used to measure the amount present. Fig. 2 shows the amount of austenite and the corresponding hardness in specimens heated 1 h at each of a series of temperatures in the range 520° to 720°C (970° to 1330°F). In this steel, A_s is about 500°C (930°F) rather than near 650°C (1200°F) as the inflection of the hardness curve would indicate. Retained austenite, unlike martensite, leads to little or no hardness increase after cooling to room temperature. It is believed that the combination of an extremely small crystal size and enrichment of the austenite with alloying elements contributes to the stability of the austenite that forms at these relatively low temperatures. As Fig. 2 shows, austenite formed above 650°C (1200°F) transforms on cooling to room

temperature. Similar results were obtained for the other steels investigated.

Effect of Cold-Work on Austenite Formation and Microstructure

If the steel is cold-worked prior to annealing in the ferrite-plus-austenite region, the rate of formation of

austenite is markedly increased, particularly at low annealing temperatures. The effect depends to a great extent on the severity of cold-work, as shown in Fig. 3 for specimens rolled at room temperature, then annealed at 500°C (930°F). Similar results were obtained by swaging at room temperature prior to annealing.

Although cold-working increases the rate of austenite formation, a more important benefit is derived from its effect on the distribution of austenite. The preferred sites for austenite formation in specimens that are not cold-worked prior to annealing are martensite plate interfaces and especially prior austenite grain boundaries. However, cold-working prior to annealing results in a uniform distribution of equiaxed ferrite and austenite. Electron micrographs of typical structures are shown in Fig. 4. Without prior cold work, Fig. 4(a), the microstructure retains the acicular appearance of the original martensite, whereas cold-working prior to annealing results in a new, equiaxed grain structure, Fig. 4(b). The phases form as extremely small crystals which grow with increasing time at temperature. However, since the temperatures are relatively low and the recrystallized structure is two-phase, grain growth is retarded and the grains remain small even after a long time at temperature. Fig. 5 shows typical structures of specimens held at 500°C (930°F) for 100, 300, 1000

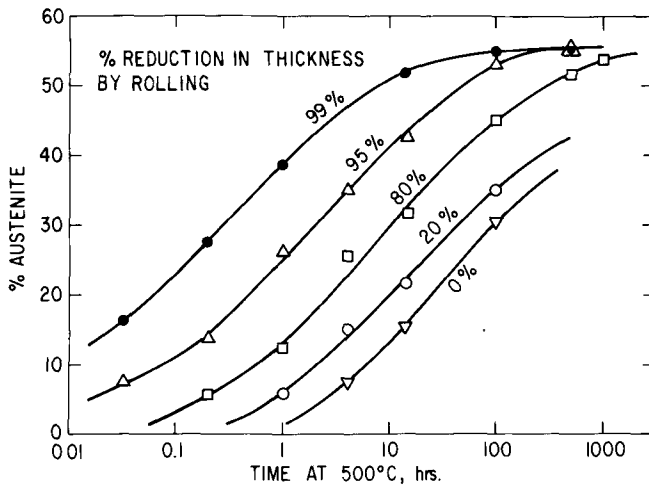
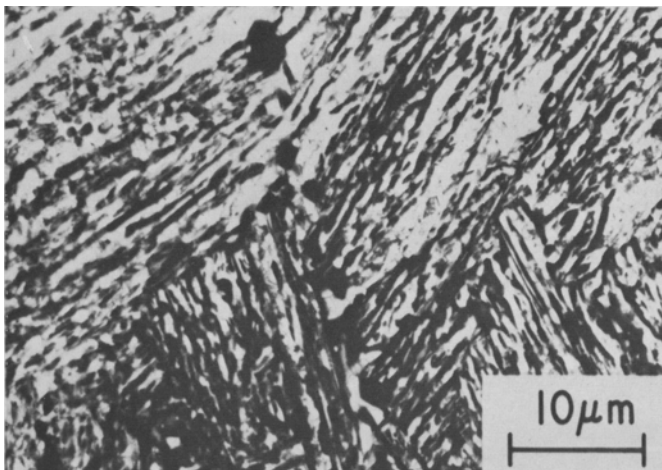
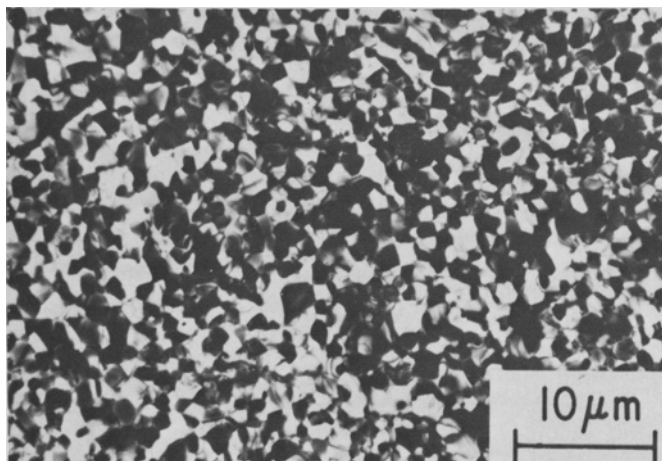
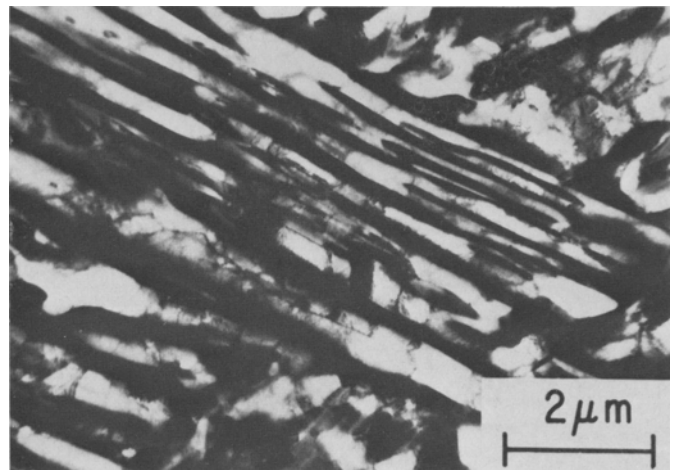


Fig. 3—Effect of cold-work on austenite formation (Steel E).



(a)



(b)

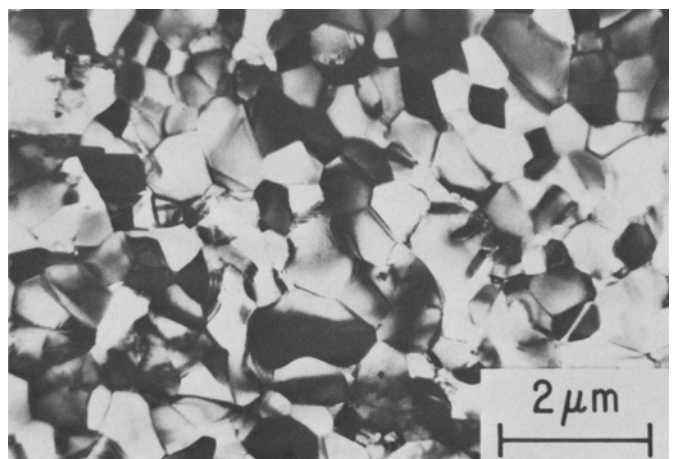


Fig. 4—Effect of prior cold-work on the annealed microstructure (low and high magnifications, Steel B). (a) Annealed 16 h, 600°C; (b) Cold-worked and annealed 16 h, 600°C.

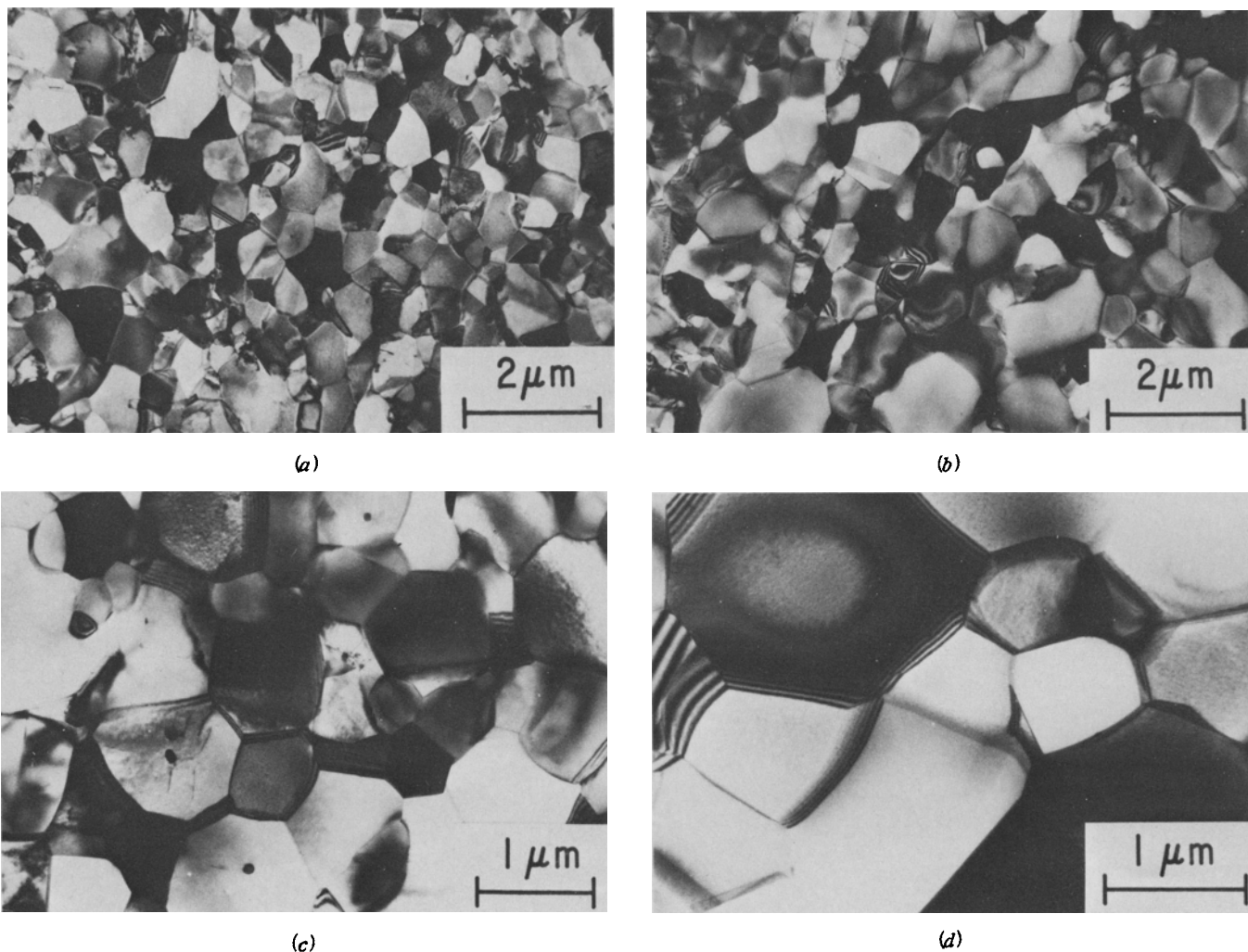


Fig. 5—Effect of annealing time at 500°C (930°F) on microstructure and grain size (Steel E). (a) 100 h; (b) 300 h; (c) 1000 h; (d) 2000 h.

and 2000 h. The grain size after 2000 h is less than 1 μm . Naturally, grain growth is slower at temperatures below 500°C (930°F) and somewhat faster at higher temperatures.

Since no transformation occurs during cooling from 500° or 600°C (930° or 1110°F), the ultrafine-grained microstructure observed at room temperature in Figs. 4 and 5 is believed to be identical to that present at the annealing temperature. Because of their small size, it is difficult to distinguish between ferrite grains and austenite grains. Occasionally annealing twins are observed in the nickel steel and stacking faults in the manganese steel which obviously identify austenite grains. As mentioned earlier, the austenite that forms during annealing at higher temperatures is less stable and transforms during cooling. Therefore, a variety of ultrafine-grained microstructures consisting of either ferrite and austenite, ferrite and martensite or ferrite, martensite, and austenite can be obtained, depending on the alloy content of the steel and the annealing temperature.

Lattice Constants and Austenite Stability

Room-temperature lattice constants of the austenite and ferrite were determined in Steel E specimens cold-

worked, then heated to 500°C (930°F) for time intervals ranging from 1 to 1000 h. No significant variation in the austenite lattice parameter with time at temperature was observed, which indicates that the first measurable amount of austenite is already enriched in nickel. The lattice constant obtained, $a = 3.5865$, indicates that the austenite which forms at 500°C (930°F) contains 30.5 at. pct Ni.¹¹ Corresponding changes in nickel content of ferrite could not be measured because the ferrite lattice constant does not change appreciably with nickel content between approximately 8 and 20 pct Ni. However, the value obtained from specimens held longest at 500°C (930°F) was $a_0 = 2.869$, which indicates that the ferrite contains approximately 6 at. pct Ni.¹² Thus, the austenite is enriched in nickel at the expense of the nickel content of the ferrite, in agreement with the phase relations in Fig. 1.

Although 30.5 pct Ni austenite with normal grain size transforms to martensite on cooling below room temperature, tests conducted on the ultrafine-grained austenite showed that no transformation occurred even after cooling in liquid helium (4 K). This additional stability is probably due to the closely-spaced boundaries which are known to inhibit transformation of austenite.¹³

Table II. Test Results

Steel	Annealing Treatment	Austenite Vol, Pct	Grain Size d , μm	$d^{-1/2}/\text{mm}^{-1/2}$	Lower Yield Stress, ksi	Ultimate Tensile Stress, ksi	Total Elongation, Pct	Uniform Elongation, Pct	Reduction in Area, Pct	
A, 9 pct Ni	1 h 565C	10	0.37	52.0	122.0	*	12.5	0	†	
					123.5	*	13	0	68.0	
	1 h 575C	12	0.44	47.7	120.4	*	17.5	0	†	
					120.9	124.9	20	12	69.6	
	4 h 575C	15	0.52	43.8	113.2	*	19	0	†	
					115.2	121.8	22.5	14	69.5	
	1 h 600C+	20	0.59	41.2	110.9	126.8	23	17	†	
	1 h 575C				112.0	131.0	25	16.5	66.7	
	16 h 575C	21	0.65	39.2	108.2	112.6	24	18	†	
					106.6	117.5	25	18	69.6	
B, 6 pct Mn	1 h 625C+	20	0.88	33.7	88.9	159.2	19.5	14	†	
	1 h 575C				89.2	161.5	18.5	13	61.8	
	1 h 560C	11	0.33	55.0	138.4	142.2	12	*	66.0	
	4 h 520C	10	0.35	53.4	135.4	138.5	19.5	*	66.4	
	1 h 600C	23	0.40	50.0	126.7	132.5	18.5	*	70.9	
	1 h 640C	30	0.45	47.1	115.7	166.0	30.5	*	49.0	
	16 h 600C	29	0.55	42.6	105.3	127.4	34	*	69.2	
	16 h 640C	30	0.90	33.3	87.0	174.0	18.5	*	20.0	
	C, 16 pct Ni	1000 h 450C	15	0.51	44.3	111.3	*	7	0	†
		100 h 500C	23	0.49	45.2	114.7	*	7	0	†
300 h 500C		26	0.59	41.2	101.2	*	10	0	†	
1000 h 500C		31	0.69	38.1	96.0	*	19	0	†	
D, 15 pct Ni	1000 h 450C	12	0.46	46.5	100.0	*	14.5	0	†	
	100 h 500C	18	0.40	50.0	99.3	*	11.5	0	†	
	300 h 500C	22	0.54	43.0	90.0	*	23	0	†	
	1000 h 500C	25	0.77	36.0	82.5	85.5	26	22.5	†	
	16 h 500C	41	0.33	55.0	137.0	*	7	0	†	
E, 21 pct Ni	100 h 500C	53	0.37	52.0	121.5	*	7	0	†	
	300 h 500C	54	0.49	45.2	109.5	*	10.5	0	†	
	1000 h 500C	54	0.67	38.6	98.5	*	24	0	†	
	300 h 500C	46	0.57	41.9	105.8	*	20	0	85.1	
	500 h 500C	48	0.66	38.9	104.5	106.1	24	16	85.2	
	1000 h 500C	51	0.70	37.0	93.0	96.3	27.5	18	85.9	
	2000 h 500C	54	0.98	31.9	85.9	94.0	29	22.5	84.1	
4000 h 500C	54	1.10	30.1	81.6	91.2	31	24	82.1		

*Indicates value was not obtained.
†Indicates flat tensile specimens.

Effect of Grain Size on Strength

To demonstrate the strength-grain size relation in the submicron grain size range, tensile specimens were prepared from the 21 pct nickel steel processed to obtain a wide range in grain size. Cylindrical tensile specimen blanks from cold-swaged material and flat tensile specimen blanks from cold-rolled material were annealed at 500°C (930°F) for time intervals from 16 to 4000 h. These treatments were selected because they produce essentially 50-50 mixtures of ferrite and austenite and the austenite has sufficient stability to resist transformation during straining. The grain sizes ranged from 0.33 to 1.1 μm and the austenite content from 41 to 54 volume pct. Tensile test results, grain sizes and austenite contents are listed in Table II. The lower yield stresses plotted against the inverse of the square root of grain diameter in Fig. 6 give a reasonably good linear relation. The slope of the line, k in Eq. [1], is 2150 psi/mm^{-1/2} (1.51 kg/mm^{3/2}). This slope is between the value reported for ferritic steel,² $k = 2630$ psi/mm^{-1/2} (1.84 kg/mm^{3/2}), and the value for austenitic steel,⁴ $k = 1770$ psi/mm^{-1/2} (1.24 kg/mm^{3/2}).

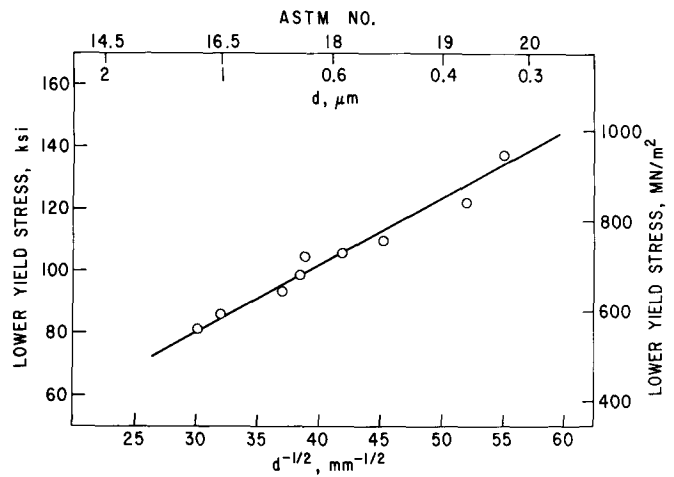


Fig. 6—Grain size-stress relation in the submicron grain-size range (Steel E).

A similar grain size-strength relation was observed in all the ultrafine-grained steels investigated, with the exception of a low-carbon steel. Fig. 7 compares

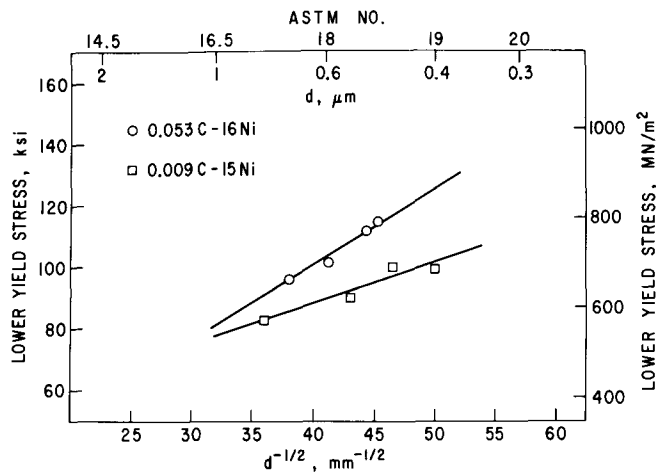


Fig. 7—Effect of carbon content on the grain size-stress relation in the submicron grain-size range.

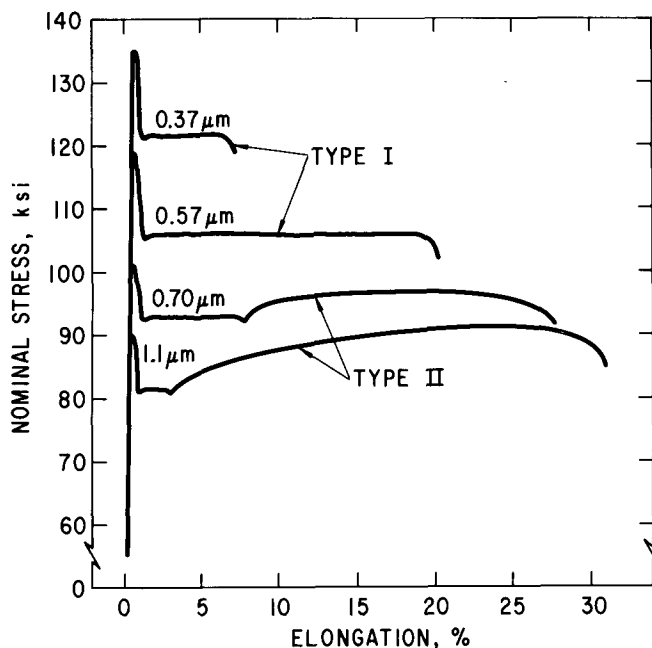


Fig. 8—Two types of stress-elongation curves and the effect of grain size.

the results of two nickel steels with different carbon contents. In the steel with 0.053 pct C, the slope, k , is $2470 \text{ psi/mm}^{-1/2}$ ($1.74 \text{ kg/mm}^{3/2}$) whereas with 0.009 pct C, k is only $1330 \text{ psi/mm}^{-1/2}$ ($0.93 \text{ kg/mm}^{-3/2}$).

This difference in slope is believed to be related to the carbon content and more specifically, to the role that carbon and other interstitial elements play in strengthening grain boundaries. The effect of carbon on the strength-grain size relation has been observed through studies in which specimens are either decarburized or heat-treated to remove the influence of carbon.¹ However, since grain-boundary area increases as the grain size decreases, it should be possible to produce a similar effect by maintaining a constant carbon content and reducing the grain size several orders of magnitude. McLean's analysis¹⁴ shows that the concentration of carbon in grain boundaries, assumed to be three atoms in thickness, is about 0.0015 wt pct when the grain size is $10 \mu\text{m}$ but this increases to about 0.015 wt pct when the grain size is reduced to

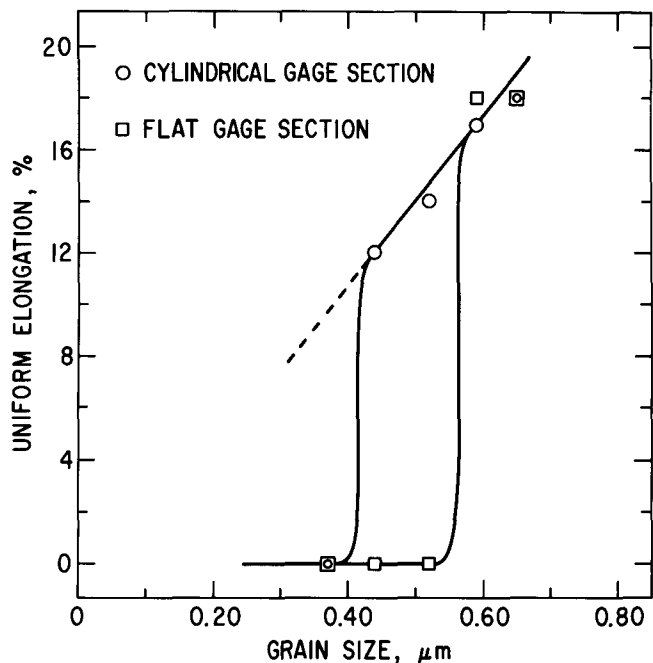


Fig. 9—Effect of grain size on uniform tensile elongation in flat and cylindrical specimens (Steel A).

$1 \mu\text{m}$. In the submicron grain size range, an even greater amount of carbon would be required to saturate the grain boundaries. It is likely that the strength of Steels C and D would be similar in the grain size range above $1 \mu\text{m}$ but they diverge, as shown in Fig. 7, because Steel D has insufficient carbon to completely strengthen the grain boundaries. Such a discontinuity in the Hall-Petch plot due to impurities has been predicted by Li¹⁵ and others but has not been demonstrated in steel of normal purity.

Effect of Grain Size on Tensile Elongation

Stress-strain curves exhibited a well-defined upper yield point followed by localized deformation at constant stress, similar to Lüders deformation. This type of yielding, which is characteristic of low-carbon steel, was observed in all the steels investigated regardless of the relative proportions of ferrite and austenite in the microstructure. However, plastic instability was observed in several of the steels. This instability is characterized by necking in the deformation band before the band traverses the entire gage section of the specimen. As a result, there is no uniform elongation and a very small total elongation.

Typical stress-elongation curves for these two types of specimens are shown in Fig. 8, designated as Type I and Type II. When the grain size is $0.7 \mu\text{m}$ or larger, deformation at constant stress (Lüders deformation) is followed, as in the normal case by a rise in stress (Type II) and total elongation values near 30 pct are obtained. However, when the grain size is $0.57 \mu\text{m}$ or smaller, the specimens neck and fracture during Lüders deformation (Type I) and total elongation values are as low as 7 pct.

Such behavior, not usually encountered in normal strain-rate, room-temperature tests, is attributed to the exceptionally small grain size. For polycrystalline specimens deforming homogeneously, true stress, σ , is empirically related to true strain, ϵ , by

$$\sigma = K\epsilon^n \quad [2]$$

where K is a constant and n is the strain-hardening exponent. In a uniaxial tensile test, the specimen begins to neck when $\epsilon = n = \epsilon_u$, the uniform strain. If deformation occurs nonhomogeneously, such as in Lüders deformation, the specimen necks when

$$\epsilon_L > n \quad [3]$$

where ϵ_L is the Lüders strain. Various factors such as strain rate, test temperature, carbon content and grain size^{2,16} are known to affect ϵ_L and n (or ϵ_u). Refining the grain size causes ϵ_L to increase and n to decrease; therefore the unstable condition represented by Eq. [3] can be created by changing only one variable, the grain size. This is in agreement with the observations in Fig. 8 and with previous observations on a ferritic steel.¹⁶

The effect of grain size on Lüders strain can be seen in Fig. 8. Lüders deformation is complete after about 3 pct elongation when the grain size is 1.1 μm and after about 8 pct when the grain size is 0.70 μm . Lüders strain in the 0.57 μm grain-size specimen appears to be greater than 20 pct; however, the specimen necked before Lüders deformation was complete. Necking began soon after the deformation band formed when the grain size was 0.37 μm . The increase in ϵ_L and the decrease in n with decreasing grain size is evident in the two Type II curves in Fig. 8.

In addition to the effect of grain size, results from several steels indicate that specimen geometry also affects Lüders band necking, Fig. 9. When plastic instability occurs, uniform elongation drops to zero. In the 9 pct Ni steel, this occurs in the flat tensile specimens between grain sizes of 0.52 and 0.59 μm and in the cylindrical tensile specimens between 0.37 and 0.44 μm . This effect is believed to be related to the sharpness of the strain gradient during Lüders deformation. In flat specimens, the deformation front is more clearly defined and the strain gradient steeper than in cylindrical specimens, probably because of less restriction to the formation of the deformation band in flat specimens.

Effect of Grain Size and Austenite Stability on Other Tensile Properties

The effect of grain size on yield strength and tensile elongation has been discussed. Because of plastic instability in ultrafine-grained specimens, a decrease in grain size can result in a decrease in total elongation and a very abrupt decrease in uniform elongation. When this occurs, the steel does not strain harden and therefore the yield strength equals the ultimate tensile strength. Reduction in area is not affected by plastic instability, however, as shown in Fig. 10. Reduction in area in all the steels investigated was essentially unchanged over a fairly wide grain size range, although some previous results indicate that it is improved by grain refinement.¹ A slightly smaller reduction in area at larger grain sizes is believed to result from transformation of austenite to martensite during straining. Examination by X-ray diffraction of fractured tensile specimens of Steels A and B verified that such transformation had occurred. Frequently this process produces serrations in the load-elongation curve.

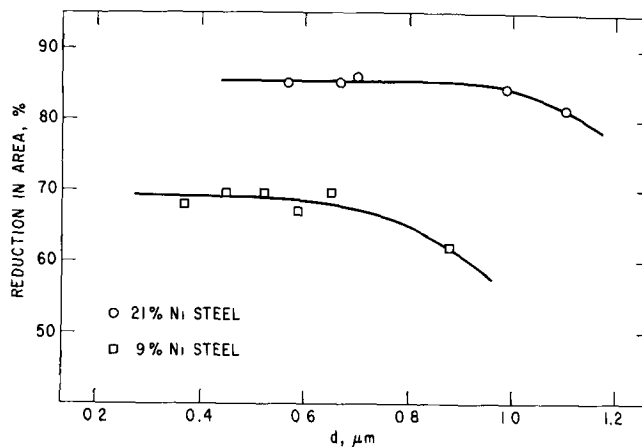


Fig. 10—Effect of grain size on reduction in area.

Accompanying the strain-induced transformation of austenite, a corresponding rise in ultimate tensile stress is observed in several 9 pct Ni and 6 pct Mn specimens, Table II. Austenite becomes less stable by increasing the grain size (longer annealing time) or by reducing the degree of alloy partitioning to the austenite (higher annealing temperature). Transformation of austenite during straining results in a Type III deformation, with a high rate of work-hardening, shown in Fig. 11, in contrast to Types I and II discussed earlier. Therefore, by controlling the annealing conditions, it is possible to prevent early necking and to obtain a more desirable type of plastic behavior (Type III) rather than very low strain hardening (Type II) or plastic instability (Type I), both of which are characteristic of ultrafine-grained specimens.

Warm-Working

The ultrafine-grained microstructure obtained by cold-working and heating at temperatures in the two-phase region can also be produced by working the steel at the annealing temperature. Specimens were warm-worked as described earlier. As Fig. 12 shows, warm-working the steel has nearly the same effect on austenite formation as cold-working-plus-annealing in the $\alpha + \gamma$ region. Essentially the same uniformly fine distribution of austenite and quite similar mechanical properties can be obtained by this alternate procedure.

SUMMARY

An ultrafine-grained microstructure can be obtained by annealing cold-worked alloy steels at temperatures in a two-phase, $\alpha + \gamma$ region. Heating at these temperatures produces equiaxed grains of low-alloy ferrite and high-alloy austenite. Since the annealing temperatures are relatively low and the recrystallized structure is two-phase, grain growth is restricted and the grain size remains small even after a long holding time. Specimens with grain sizes in the range 0.3 to 1.1 μm (ASTM 20 to 16) were obtained in manganese and nickel steels by annealing for 1 to 4000 h at temperatures between 450° and 650°C (840° and 1200°F).

The well-established grain size-strength relation is observed in the submicron grain-size range. In a 21 pct Ni steel, for example, decreasing the grain size from 1.1 to 0.33 μm results in a 68 pct increase in

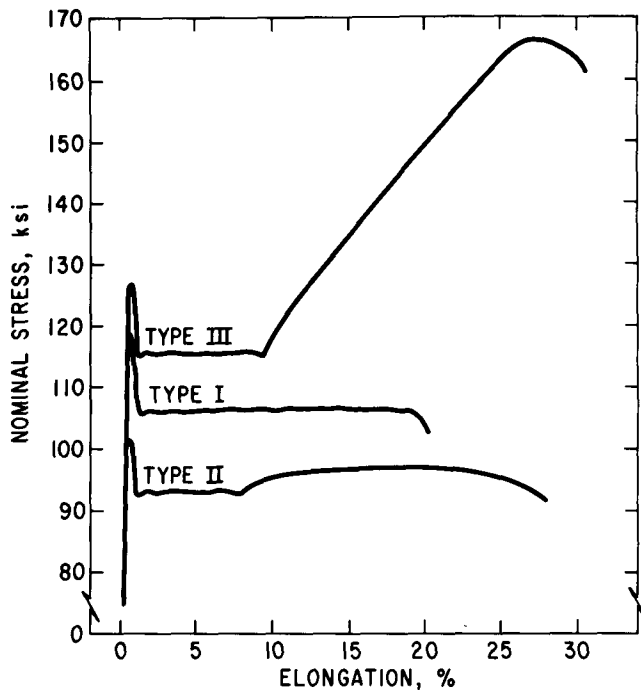


Fig. 11—The three types of stress-elongation behavior observed in ultrafine-grained specimens.

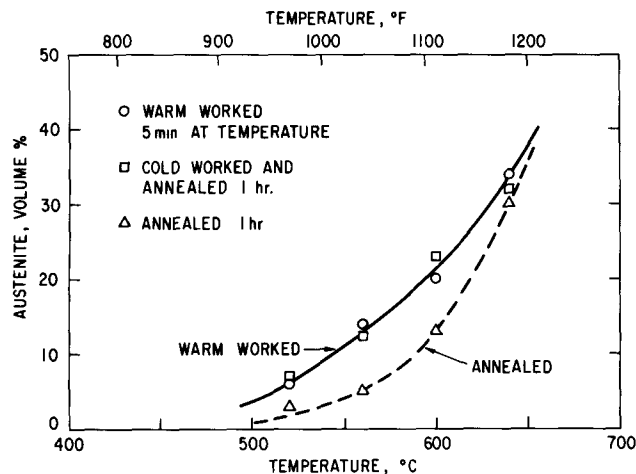


Fig. 12—Effect of warm-working on the formation of austenite (Steel B).

yield stress. The grain size dependence of the lower yield stress, k , is $2150 \text{ psi/mm}^{-1/2}$ ($1.51 \text{ kg/mm}^{3/2}$), which is between the values reported for ferritic and austenitic steels.

A similar grain size-strength relation was observed in all but one of the steels investigated. The value of k is much lower in this steel probably because its lower carbon content provides insufficient carbon to fully strengthen the grain boundaries. Carbon and other surface-active elements are believed to migrate to grain boundaries and the total solubility of such impurities increases greatly when the grain size is reduced several orders of magnitude. Thus, normal impurity contents may be adequate to strengthen boundaries

throughout the normal grain size range, but not in the submicron range.

Depending on various factors such as grain size, austenite stability and specimen geometry, three distinct types of plastic behavior were observed in ultrafine-grained, ferrite-austenite specimens. All tensile specimens yielded with a well-defined upper yield point followed by localized deformation at constant stress, similar to Lüders deformation. With the smallest grain sizes, specimens necked within the deformation band and fractured before the band traveled the length of the gage section. This results in very low elongation values because most of the deformation occurs only in the necked portion of the specimen. In slightly larger grain size specimens, deformation at constant stress is followed by a slight rise in stress in the normal manner. A third type of behavior was observed in specimens in which the austenite partially transformed to martensite during straining. This transformation prevents early necking and greatly increases the strain-hardening capacity of the steel. Thus, a low rate of strain hardening and plastic instability, both of which are characteristic of ultrafine-grained steels, can be avoided by controlling the resistance of the austenite to transformation. Austenite is made less stable by increasing the grain size or by reducing the degree of alloy partitioning.

Essentially the same ultrafine-grained microstructure can also be obtained by warm-working at temperatures in the $\alpha + \gamma$ region. By controlling the conditions for annealing and/or warm-working, various combinations of ultrafine-grained ferrite and austenite, ferrite, and martensite, or ferrite, austenite, and martensite can be obtained.

ACKNOWLEDGMENTS

The author gratefully acknowledges helpful discussions with W. B. Morrison, R. A. Grange, and W. C. Leslie and the assistance of C. E. Groff, R. C. Glenn, K. R. Phillips, and W. B. Seens, his associates at the U. S. Steel Corporation Research Laboratory.

REFERENCES

1. R. W. Armstrong: *Advan. Mater. Res.*, 1970, vol. 4, p. 101.
2. W. B. Morrison: *Trans. ASM*, 1966, vol. 59, p. 824.
3. R. A. Grange: *Trans. ASM*, 1966, vol. 59, p. 26.
4. W. B. Morrison and R. L. Miller: *Ultrafine-Grain Metals*, Proc. 16th Sagamore Conf., p. 183, Syracuse Univ. Press, 1970.
5. R. W. Armstrong: *Ultrafine-Grain Metals*, Proc. 16th Sagamore Conf., p. 1, Syracuse Univ. Press, 1970.
6. L. F. Porter and D. S. Dabkowski: *Ultrafine-Grain Metals*, Proc. 16th Sagamore Conf., p. 133, Syracuse Univ. Press, 1970.
7. E. A. Owen and Y. H. Liu: *J. Iron Steel Inst.*, 1949, vol. 163, p. 132.
8. R. L. Miller: *Trans. ASM*, 1968, vol. 61, p. 592.
9. R. C. Glenn and R. D. Schoone: *Rev. Sci. Instrum.*, 1964, vol. 35, p. 1233.
10. J. E. Hilliard: *Metal Progr.*, May 1964, vol. 85, p. 99.
11. W. B. Pearson: *Handbook of Lattice Spacings and Structures of Metals and Alloys*, vol. 1, Pergamon Press, New York, 1958.
12. L. Zwell, D. E. Carnahan, and G. R. Speich: *Met. Trans.*, 1970, vol. 1, p. 1007.
13. W. C. Leslie and R. L. Miller: *Trans. ASM*, 1964, vol. 57, p. 972.
14. D. McLean: *Grain Boundaries in Metals*, p. 130, Oxford University Press, London, 1957.
15. J. C. M. Li: *Trans. TMS-AIME*, 1963, vol. 227, p. 239.
16. W. B. Morrison: 2nd Int. Conf. on *The Strength of Metals and Alloys*, vol. III, p. 879, ASM, 1970.