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The Effect of Grain Size on the Shock-Loading Response of 304-Type Stainless Steel

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The residual microstructure and mechanical response of shock-loaded stainless steel (AISI-304) of four different grain sizes – 23, 55, 85 and 187 μm – was investigated. In addition to mechanical twinning and planar dislocation arrays, transformation to both ϵ and α martensite occurred in all shock-loaded specimens but became more extensive with decreasing grain size. In comparison to the Hall-Petch behavior of yield and early flow stress observed for the material after 5.2 pct cold rolling, the strengthening efficiency of shock loading decreased with increasing grain size. Shock loading enhanced the strain-induced transformation to α martensite during subsequent tensile deformation.

THE interactions of plastic waves with metals are a well known phenomenon.[1,2] Shock waves can produce one or a combination of the following metallurgical effects: generation of point defects,[3] dislocations and twins,[4] phase transformations and precipitation.[5] The two unique features of shock loading that differentiate it from quasistatic deformation are the high strain rates ($10^4 - 10^6$ s^{-1}) and the small residual strains introduced.

Although the response of a variety of metals to shock waves has been investigated, little effort has been concentrated on systematically studying the effect of metallurgical variables. It seems, however, that the strengthening effect due to shock loading may be altered by structural variables, for example by precipitation (Nordstrom *et al*,[6] Meyers and Orava[7]). One of the most important structural parameters which affect mechanical properties is grain size. Jones and Holland[8] found no measurable effect of grain size on the Hugoniot elastic limit (dynamical yield strength) during explosive loading of mild steel. However, these authors did not investigate the residual defect structure which is directly responsible for the strengthening effects often observed after shock loading. It was thus the objective of the present investigation to study the effect of grain size on the microstructure and mechanical properties of shock-deformed 304 stainless steel. The response was compared to that of material conventionally deformed by cold rolling.

EXPERIMENTAL PROCEDURE

Sheet samples of type 304 stainless steel (3.2 mm thick) of composition shown in Table I were heat treated at various temperatures in protective "Sentripak" bags or under vacuum in order to produce the desired grain sizes, and quenched in oil to prevent sensitization. Heat treatments were chosen in such a way as to provide the widest possible range of grain sizes consistent with the sheet dimensions and initial grain size. Four different sizes were obtained by proper heat treatments. The average grain diameters, as measured by the linear intercept method, were: 23, 55, 85 and 187 μm.

The shock loading assembly is shown in Fig. 1. A "mousetrap" type system was used. The dimensions of the various components were calculated according to Orava and Wittman.[9] The main charge consisted of a cast Pentolite slab. The plane-wave generator was made of wax-imbedded RDX sandwiched between two 3 mm thick glass plates. The line-wave generator was formed with six pieces of Primacord having equal length. The six sections were arranged in such a way as to have one bound extremity (for detonator insertion) and the other penetrating about 3 mm into the sandwiched assembly. A copper driver plate (6.4 mm thickness), accelerated to a velocity of 530 m/s, provided a pressure of 10 GPa (100 Kbar) and pulse duration of approximately 2.8 μs at the samples. Upon detonation the whole assembly was accelerated into a water tank with a sand bottom. One protective cover plate and one sample plate with the same thickness (3.2 mm) were used for each experiment; both were of the same 304 stainless steel, having undergone the same heat treatment. Consequently, they exhibited the same grain size. The smooth surfaces of the recovered driver plates and cover plates showed that the modified line-wave generator used in the experiments had a satisfactory performance. This was confirmed by the regularity in hardness of the sample plates.

The material processed by shock waves was compared to material conventionally deformed by cold rolling. Cold rolling was performed at ambient temperature by a succession of passes, with approximately 0.5 pct reduction per pass. The rolling was conducted along the same orientation as the original rolling direction of the sheet. The comparison of shock and conventionally processed material was effected at an equivalent shear strain. This is thought to be a better basis of comparison than the commonly

Table I. Chemical Composition of Alloy, Wt Pct

Ni	Cr	Mn	Si	Mo	C*	Fe
9.95	18.50	1.45	0.50	0.22	0.10	Balance

*Carbon content by chemical analysis higher than nominal composition of 304 SS (maximum 0.08 pct).

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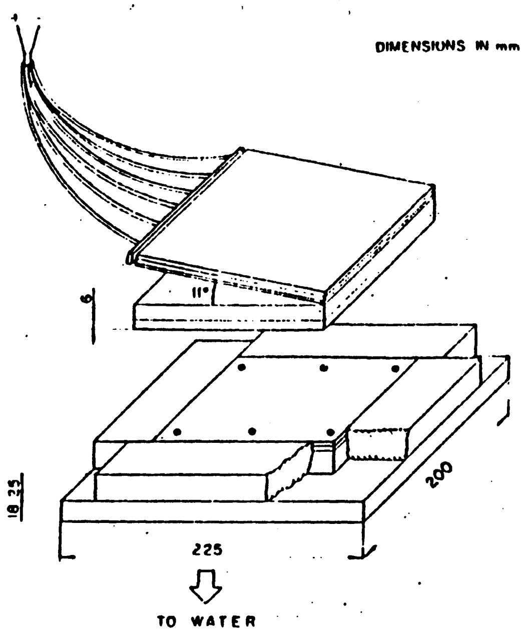


Fig. 1—Shock-loading assembly.

used effective (or generalized) strain.[7] A pressure of 100 Kbar (10 GPa) corresponds to a 5.2 pct reduction in thickness, on the basis of equal shear strains.

Tensile tests were conducted at ambient temperature in air with a floor-model 10,000 Kg capacity Instron testing machine at a nominal strain rate of 2.8×10^{-3} s^{-1}. The tensile specimens had a gage width of 4 mm, length of 30 mm and thickness of 3.2 mm. All specimens had their longitudinal axis along the original rolling direction of the sheet.

For transmission electron microscopy, in a JEOL JEM-100B electron microscope operated at 100 KV, 3 mm disks were obtained parallel to the surface of the sheets. They were cut from the central portion of the sheets by spark erosion and planing in a Servomet SMD apparatus and thinned by conventional techniques.

The X-ray identification of α and ϵ martensite was made with a Philips diffractometer using Mo Kα incident radiation.

RESULTS AND DISCUSSION

Microstructure

Due to a relatively low stacking-fault energy, the microstructure of shock-loaded austenitic stainless steels exhibits planar dislocation arrays accompanied by plate-like features on {111} austenite planes generally identified as deformation twins.[10] Metallographic observations by optical and transmission electron microscopy confirmed the typical morphology of this microstructure in shock-loaded samples of all four grain sizes, Fig. 2 and Fig. 3. In contrast, only a small number of plates were found in isolated areas of the cold-rolled samples, Fig. 4. This observation agrees with the well-established dependence of mechanical twinning on the rate of strain.[11]

Although not always evident in Fig. 2, the metallographic surfaces of all shock-loaded samples showed large variations in apparent plate density from grain to grain. Partly, these variations may be explained by the effect of plate orientation on the etching characteristics of the grain surface, but genuine variations in microstructure due to grain orientation, typical for uniaxial loading in tensile tests,[12,13] may also be expected to occur after the passage of planar shock waves, and have in fact been observed in iron shock-loaded at pressures above 13 GPa[14] and by the recovery behavior of 304 stainless steel shock-loaded to very high pressure.[15] Due to the statistically small number of grains encountered in transmission electron microscopy for any given foil orientation, no quantitative measurements were attempted. However, when micrographs of the same foil orientation were compared qualitatively, no systematic changes of plate density or dislocation substructure could be observed as a function of grain size, Fig. 3.

The shock-induced transformation to α (essentially BCC) or ϵ (HCP) martensite in metastable austenitic stainless steels such as AISI-304 still appears to be somewhat controversial.[2,11] Crystal structure analysis by electron and X-ray diffraction techniques was therefore carried out to identify a possible effect of grain size on the phases present after shock loading. The transformation to α martensite down to relatively small volume amounts (below X-ray diffractometer sensitivity) was also monitored by checking semiquantitatively the residual ferromagnetism of shock-loaded and cold-rolled samples before and after tensile testing. Table II gives a summary of the results.

A rapid electron diffraction technique[16] for the distinction of thin microtwins from morphologically identical HCP plates in an FCC matrix allowed a large number of plates to be analyzed *in situ* in the microscope. The technique simply consists of rotating the sample into one of several preestablished exact matrix orientations in which twin or HCP spot positions can be distinguished and identified by inspection. Evidence for the presence of hexagonal martensite was found in all shock-loaded samples. In some cases, ϵ plates were seen to coexist with deformation twins in

Table II. Crystal Structure Analysis in Shock-Loaded and Cold-Rolled AISI-304 of Different Grain Sizes

Experimental Technique	Treatment	Grain Size in μm		
		23	85	187
Electron diffraction	Shock loaded	Twins, ϵ frequent, α needles and plates	Twins, some ϵ, α needles	Twins, some ϵ, α needles
X-ray diffraction	Shock loaded	γ, ϵ, α	γ	γ
Magnetic techniques	Only shocked	Ferromagnetic		Very weakly ferromagnetic
	Shocked + ruptured in tension	Strongly ferromagnetic		Ferromagnetic
	Only cold rolled	No measurable ferromagnetism		No measurable ferromagnetism
	Cold rolled + ruptured in tension	Weakly ferromagnetic in necked region		Weakly ferromagnetic in necked region

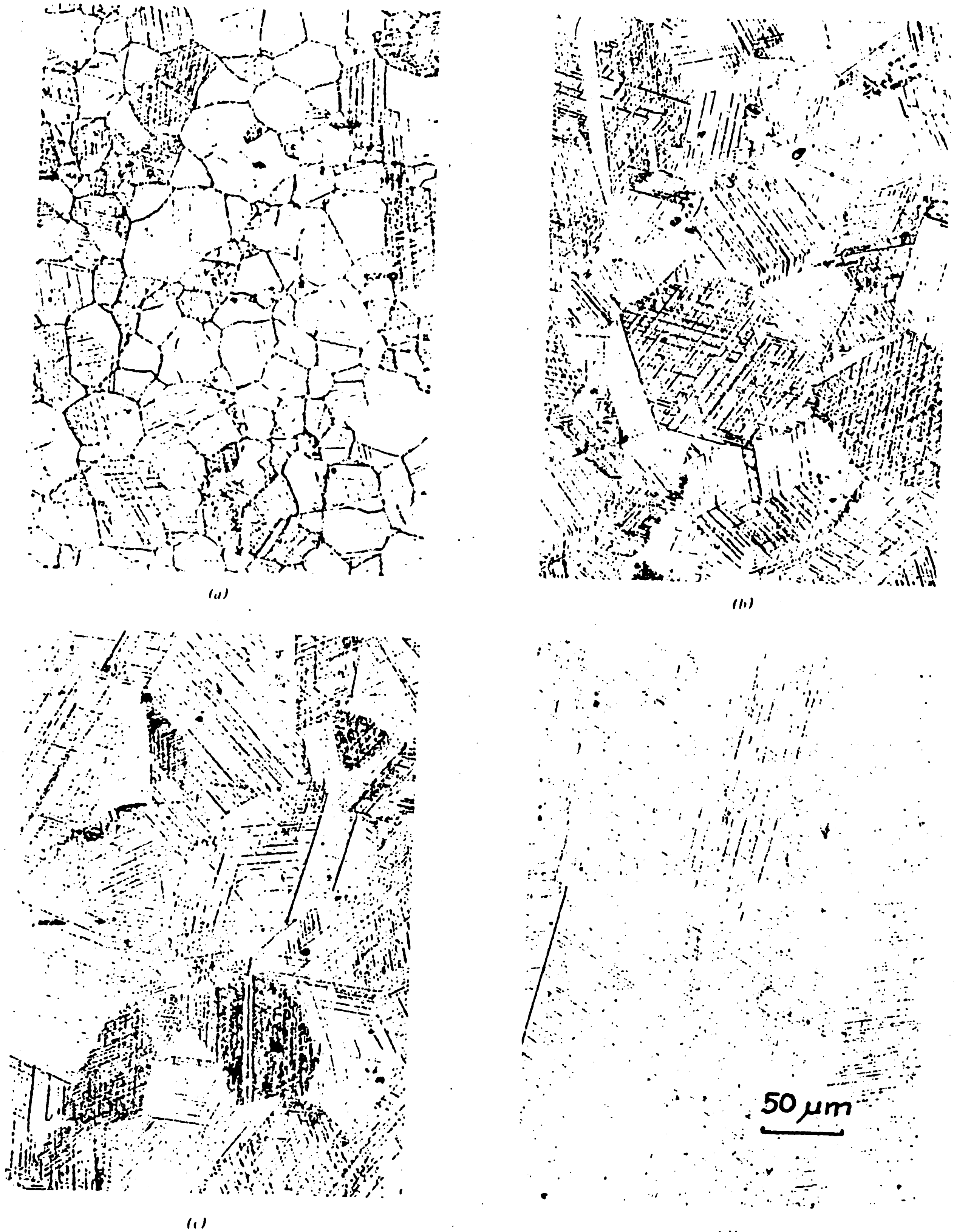
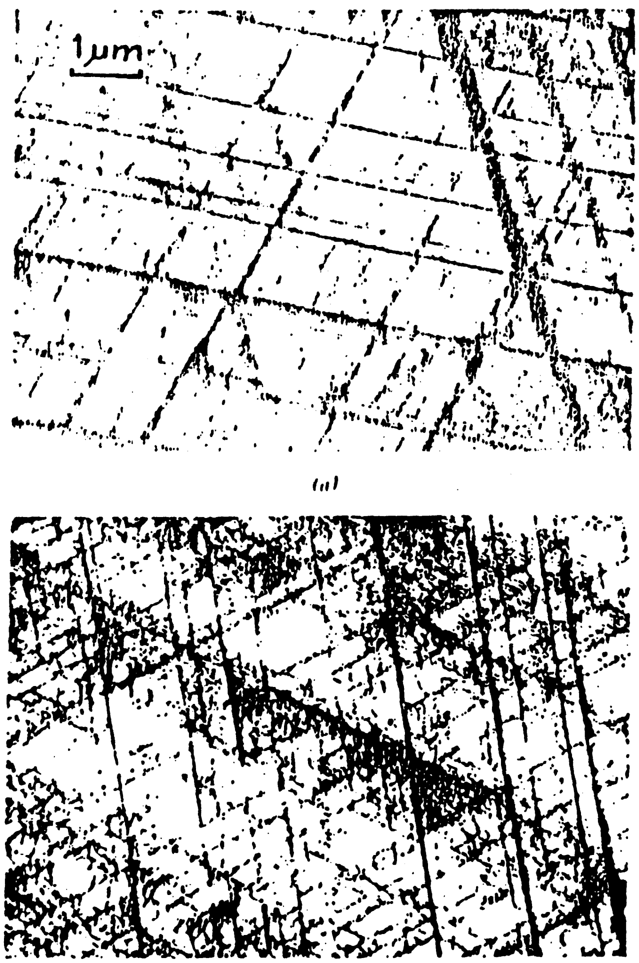


Fig. 2—Typical microstructures after shock loading as a function of grain size; (*a*) 23 μm, (*b*) 55 μm, (*c*) 85 μm, and (*d*) 187 μm.

the same grain, Fig. 5. However, while mechanical twinning appeared to be dominant for the larger grain sizes, ε plates were observed frequently for the smallest grain size, in agreement with X-ray diffraction results which presented clear intensity peaks of hexagonal positions only for the 23 μm grain size sample.



(a)

(b)

Fig. 3—Typical microstructure after shock loading as seen by transmission electron microscopy. Grain size is 23 μm in (a), and 187 μm in (b). Direction of shock wave propagation and of incident electron beam is ⟨110⟩ in both micrographs.

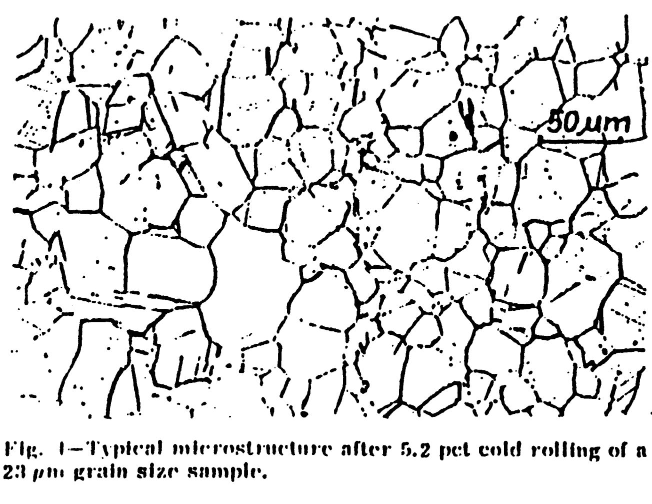


Fig. 4—Typical microstructure after 5.2 pct cold rolling of a 23 μm grain size sample.

By metallographic observations, small amounts of α martensite were found in all shock-loaded samples, in the form of thin needles along ⟨110⟩ intersections between deformation twins, ε plates or activated slip planes. Fig. 6 shows the typical morphology of this form of α martensite in optical and electron micrographs first described by Lagneborg.[17] In the 187 μm grain size sample, this α martensite could not be detected by X-ray diffraction, but produced a weak ferromagnetic response. On the other hand, BCC peaks appeared in the X-ray diffraction data of the 23 μm grain size specimen. These α martensite peaks were apparently generated in those regions where extensive martensite plates had formed during shock loading, Fig. 7. In agreement with the X-ray results, α plate martensite was only found in the 23 μm grain size sample.

Qualitatively, the same effect of grain size on the transformation to α martensite during shock-loading was also noticed when observing the samples in a magnetic field, see Table II. The magnetic response of the shock-loaded samples, much weaker for the large grain size, should be compared to no measurable amounts of ferromagnetism in any of the cold rolled specimens. After tensile test rupture, shock-loaded samples were strongly ferromagnetic, the effect again being much larger for the smallest grain size. Only weak traces of ferromagnetism within the necked region were found for the cold rolled specimens after rupture.

Mechanical Behavior

Tensile tests were conducted for the four grain sizes, each of them in three conditions: undeformed, cold-rolled and shock-loaded. Figs. 8 and 9 show the true stress-true strain curves of typical specimens for the 23 and 187 μm grain sizes, respectively; the lines were discontinued at the onset of necking. The true stress at the onset of necking was not substantially altered in any of the four grain sizes, either by conventional or shock deformation. The shock-loaded conditions, however, exhibited consistently higher yield strengths and lower ductilities than the cold-worked ones.

The effect of grain size on yield and flow stress for all three conditions is shown in Figs. 10, 11 and 12 which represent a series of Hall-Petch plots for stresses at 0.2, 1 and 5 pct plastic strain. Within the experimental scatter in Figs. 10 through 12, the data for previously undeformed and cold-rolled specimens appear to conform to a linear Hall-Petch relationship with roughly the same slope. This behavior may be expected if cold rolling did not introduce a significant number of microstructural features which could act as new obstacles to slip and is thus in agreement with the metallographic observation presented above (Fig. 4) showing little change in microstructure.

Due to the complex substructure introduced by shock loading, the grain size dependence of yield and flow stress for these specimens can be expected to follow a more complicated relationship. While the experimental scatter in Figs. 10 through 12 would probably allow to draw straight lines through

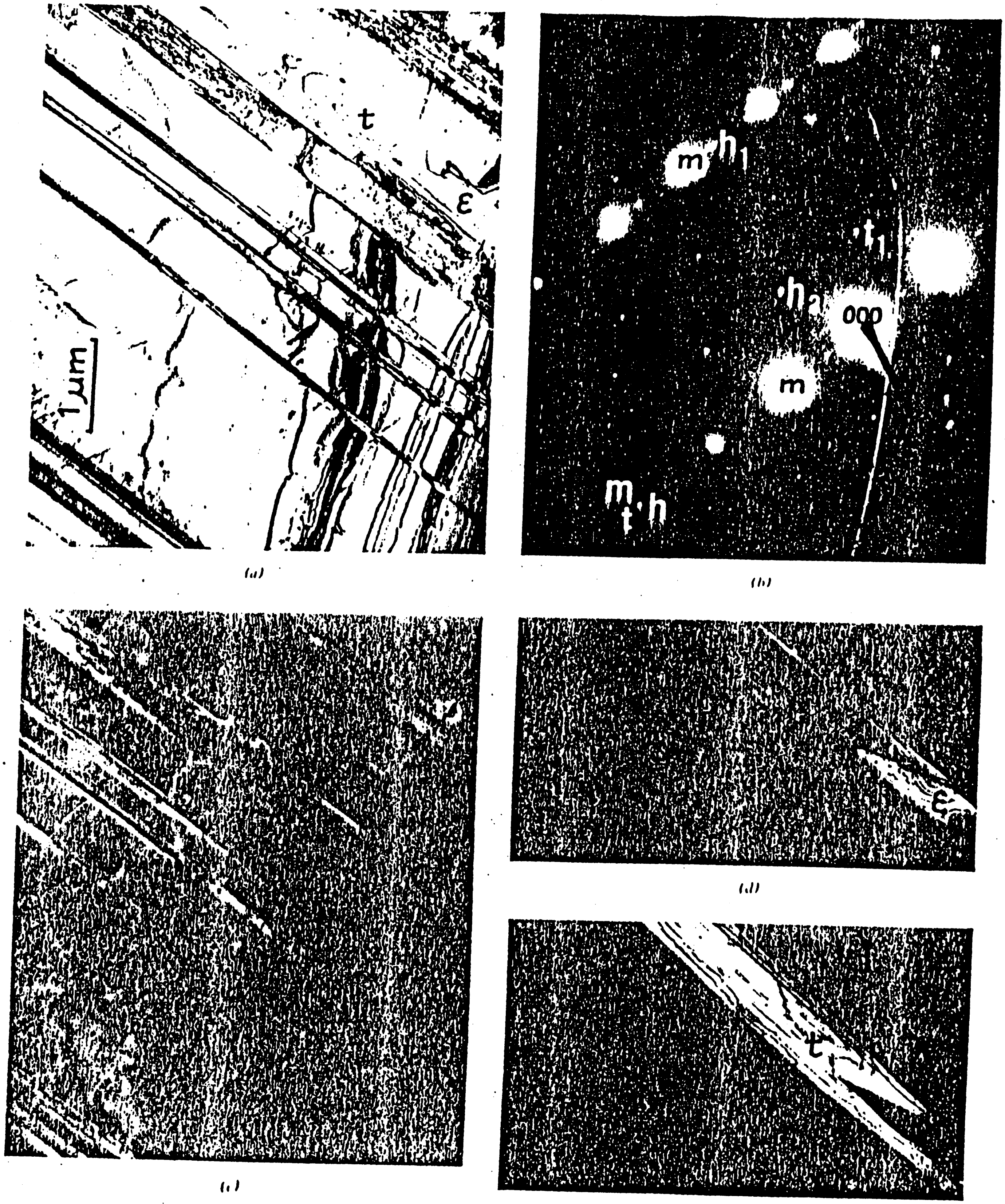
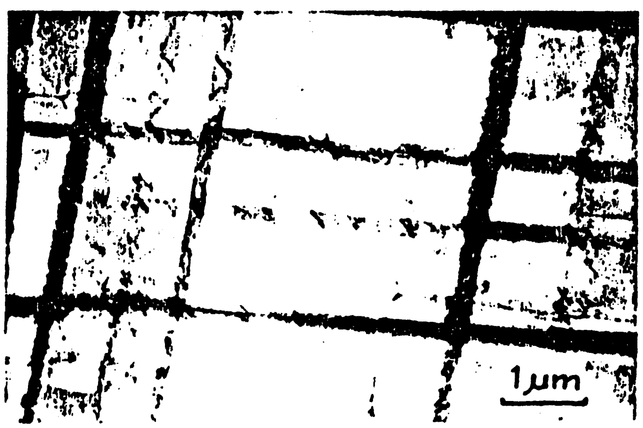


Fig. 5—Deformation twins and ε martensite after shock loading in a 187 μm grain size sample. Bright field in (*a*); diffraction pattern with matrix (*m*), twin (*t*) and hexagonal (*h*) spots in (*b*); dark field of two ε martensite habits (spot h_1 and h_2, respectively) in (*c*) and (*d*); and dark field of twin (spot t_1) in (*e*). Spot h_2 is also generated in matrix/twin interface by double diffraction, see Fig. 5(*d*).

all the data points with exception of the 0.2 pct yield stress for the largest grain size, such lines would be of doubtful value since the significant size parameter for a Hall-Petch relationship may no longer be the average grain diameter. Figs. 10 through 12 show, however, that the efficiency of shock-loading as a strengthening agent decreases with increasing grain

(a)



(b)

Fig. 6—Needle-shaped α martensite in shock-loaded AISI-304. Optical micrograph of 187 μm grain size in (a), and transmission electron micrograph of 85 μm sample in (b).

Fig. 7—Plate-like α martensite in shock-loaded AISI-304 of 23 μm grain size.

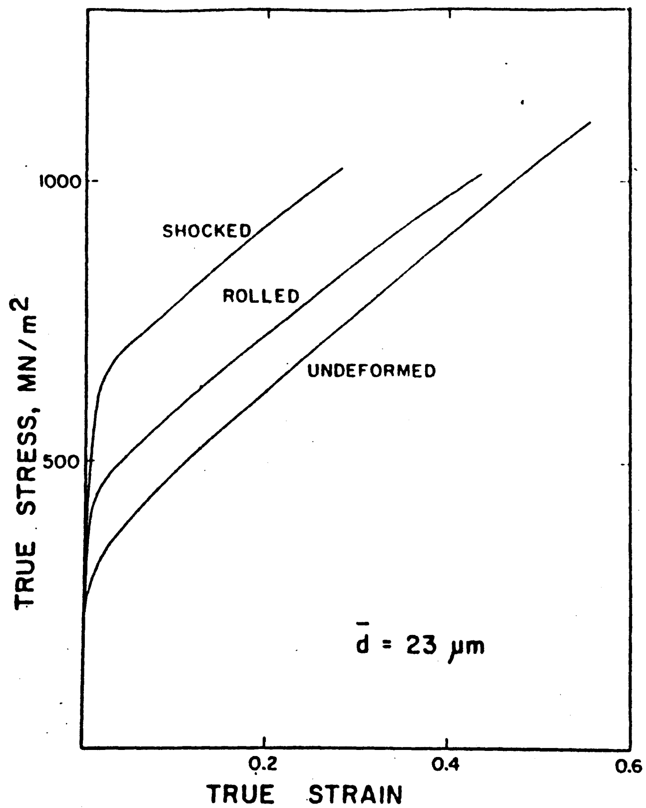


Fig. 8—True stress-true strain curves for the undeformed, cold-rolled and shocked conditions with 23 μm grain size.

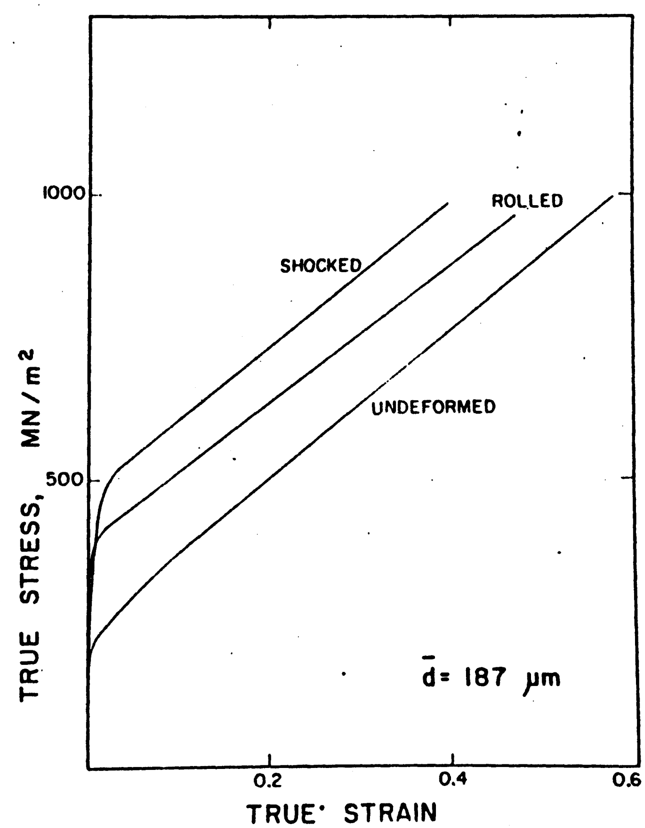


Fig. 9—True stress-true strain curves for the undeformed, cold-rolled and shocked conditions with 187 μm grain size.

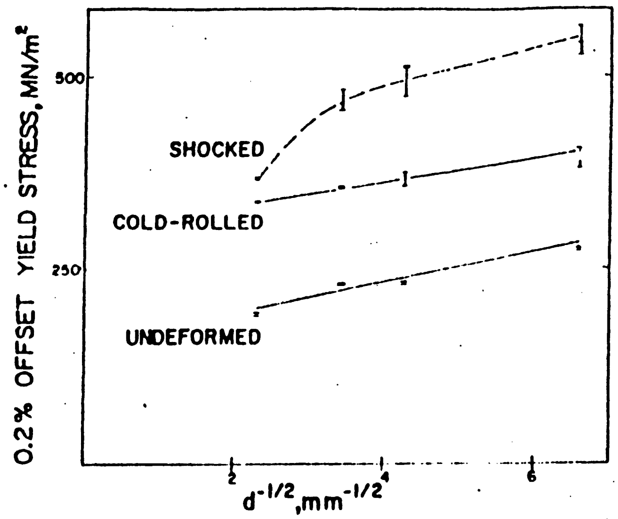


Fig. 10—Hall-Petch plot (0.2 pct offset yield stress *vs* $d^{-1/2}$) for the undeformed, cold-rolled and shocked conditions.

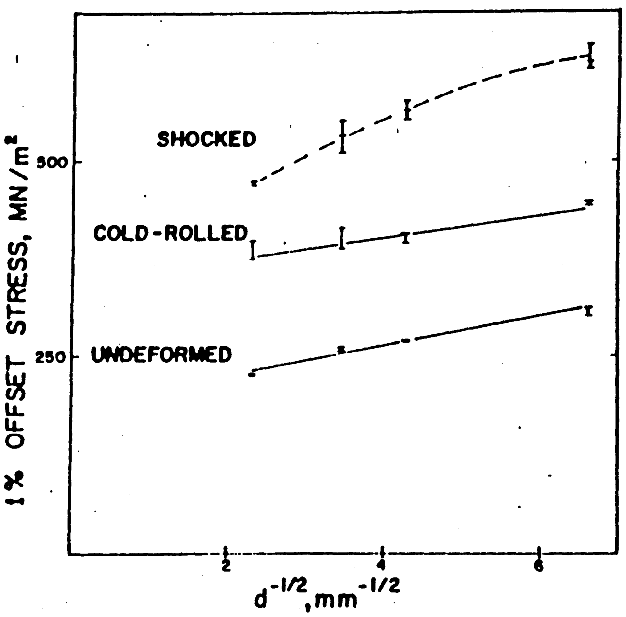


Fig. 11—Hall-Petch plot (1 pct offset stress *vs* $d^{-1/2}$) for the undeformed, cold-rolled and shocked conditions.

size. This effect appears particularly pronounced for the largest grain size and the 0.2 pct yield strength. The stress-strain curves for this grain size (Fig. 9) indicate the occurrence of some plastic flow in the shocked specimen at very low stresses, apparently below the yield stress of the cold rolled specimen, followed by rapid strain hardening. Both early yielding and rapid strain hardening could be associated to a stress-induced martensite transformation[18,19] which may produce some plastic strain in the specimen before the onset of extensive slip.

The structural observations have shown that the grain size has an effect on the residual microstructure of 304 stainless steel after shock-loading. Although this effect could not be described in quantitative terms, a gradual substitution of twins by ϵ martensite and the tendency to form more α martensite has been noted as the grain size decreases. Since it has been shown that the twinning stress, σ_t, in-

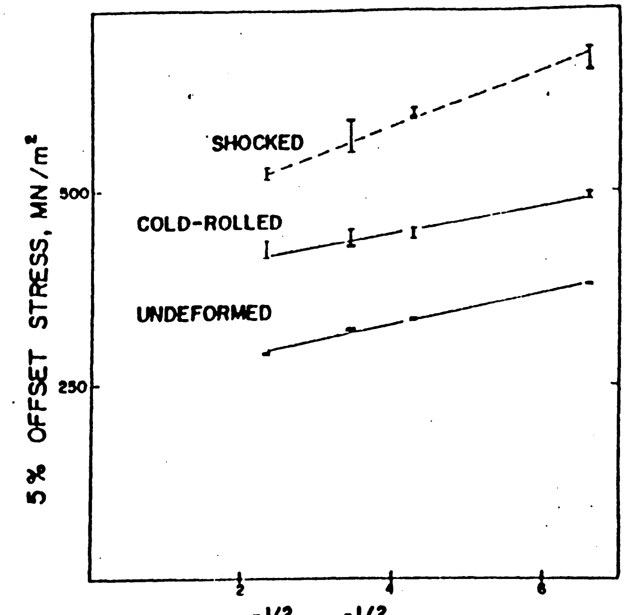


Fig. 12—Hall-Petch plot (5 pct offset stress *vs* $d^{1/2}$) for the undeformed, cold-rolled and shocked conditions.

creases with decreasing grain size,[20] the variation of α, ϵ and twins with grain sizes could be explained assuming that there also exist stresses, σ_α and σ_ϵ, necessary to start α and ϵ transformation but which decrease with decreasing grain size.* Grain size effects on the kinetics of the strain-induced martensite transformation have been observed in quasistatic deformation of 304 stainless steel,[21] and changes of the M_s temperature with grain size have been found in an Fe-Ni-C alloy.[22]

In addition, the effect of grain size on the residual microstructure may reflect an effect of grain size on the shock wave configuration. According to a "wavy wave" model recently proposed by Meyers[23,24] the shock wave in polycrystalline metals is not planar but has irregularities in the front position and peak pressure, and is affected by internal reflections. When such wave front irregularities were deliberately introduced by machining a pattern into the cover plate, the residual hardness was changed in 304 stainless steel.[25] Similarly, wave front irregularities may depend on grain size and thus lead to the different distributions of twins, α and ϵ martensite observed above.

The mechanical properties as measured by the tensile yield and flow stress must reflect the differences observed in the microstructure as a function of deformation process and grain size. Thus, the higher strength of all the shock-loaded samples with respect to their cold rolled counterparts is essentially a consequence of the criterion of the equivalent shear strain adopted in this work. Either one or all of the microstructural features involved, *i.e.* high dislocation density, high twin (or ϵ plate) density and the presence of α martensite, may have been responsible for strengthening. In the absence of quantitative information, it is difficult to assess which of these

*The authors are grateful to the reviewer who suggested this explanation.

microstructural features reduces the efficiency of shock-loading strengthening with increasing grain size. All shock-loaded samples, however, as shown by the magnetic measurements (Table II), developed considerable amounts of α martensite during tensile testing which suggests that austenite sensitization toward the strain-induced α transformation took place during shock loading. It is thought that the austenite sensitization occurred through formation of the thin α crystals lying along the intersections of twins, ϵ plates and activated slip planes. In tensile tests of austenitic stainless steels with different stacking-fault energies, larger amounts of α martensite have been observed when ϵ plates were produced during the early deformation stages, while less α martensite was present in those alloys which initially twinned.[26] Thus, austenite sensitization toward transformation to α martensite should decrease with increasing grain size and may therefore be responsible for the loss of strengthening efficiency through shock loading at larger grain sizes.

CONCLUSIONS

The principal results of this investigation may be summarized as follows:

1) ϵ and α martensite were present in all shock-loaded specimens, in addition to mechanical twinning and planar dislocation arrays. The amount of ϵ and α increased with decreasing grain size.

2) Shock loading at a pressure of 10 GPa (100 Kbar) and pulse duration of 2.8 μs increased the yield stress of 304 stainless steel substantially more than cold rolling to a reduction of 5.2 pct, in spite of the same maximum shear strains imparted by both methods of deformation.

3) In the range of grain sizes investigated (23 to 187 μm), the yield strength of annealed and cold-rolled 304 stainless steel conformed to a Hall-Petch relationship with a slope k approximately equal to 0.7 $MNm^{-3/2}$. For the shock-loaded material, the efficiency of strengthening decreased with increasing grain size.

4) Shock loading leads to austenite sensitization toward strain-induced α martensite transformation. It is suggested that this austenite sensitization occurs through the formation of small α crystals at slip intersections during shock loading.

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