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The Effect of Hydrostatic Pressure on Yielding in Iron

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ABSTRACT

Armco iron normally shows a sharp yield point in tension at atmospheric temperature and pressure. This yield is either absent or substantially reduced in specimens subjected to hydrostatic pressures of 10^3 – 10^4 atmospheres prior to test. Removal of the yield point indicates that free dislocations are created during pressurization, apparently at elastic inhomogeneities such as inclusions. Evidence is presented to support this view, e.g. the fact that high-purity iron is not greatly affected by pressure. The observations are discussed in terms of recent theories of the influence of free dislocations on yielding.

§ 1. INTRODUCTION

LARGE hydrostatic pressures applied to crystalline solids can induce phase transformations, modify defect mobilities and hence diffusion phenomena, and enable otherwise brittle materials to be deformed (Bundy *et al.* 1961, Bridgman 1949). It is now shown that pressurization at 10^3 – 10^4 atmospheres can influence the mechanical properties of metals tested subsequently at room pressure. In this and a companion paper (Bullen *et al.* 1964), the marked changes produced by pressurization on the yielding of iron and the brittleness of chromium are reported.

Evidence to be presented suggests strongly that free dislocations are created during pressurization. It is therefore appropriate at this point to consider the theory of yielding, particularly yielding in the presence of free dislocations.

§ 2. THEORY OF YIELDING

The sharp yield point in iron is due to the 'locking' of dislocations by solute atoms (Cottrell and Bilby 1949). Yielding in metals in which virtually all dislocations are locked has been extensively studied and is reasonably well understood (e.g. Cottrell 1963). Recently, quantitative theoretical treatments of yielding in the presence of small densities of free dislocations have become possible (Hahn 1962, Cottrell 1963) because of the availability of empirical data on the stresses required to drive dislocations at different velocities (e.g. Johnston and Gilman 1959, Stein and Low 1960) and on the rate of multiplication of dislocations with strain (e.g. Hahn 1962).

Basically, these treatments propose that, during yielding, the free dislocations in the specimen must move with sufficient velocity to match the applied strain rate. The stress (σ) required to produce this velocity (v) varies according to the empirical equation :

$$v = \left(\frac{\sigma}{\sigma_0} \right)^n,$$

where σ_0 is the stress for unit dislocation velocity and n is a constant (for the usual range of velocities). In iron $n \approx 35$, so that σ increases rapidly with v . As long as the number of free dislocations remains small, large stresses are necessary to produce a significant strain rate ; as the dislocations multiply, the average velocity required is less and σ decreases. The stress/strain relationship at yield is therefore a complex function of the initial density of free dislocations, their rate of multiplication, the particular value of n and the rate of hardening with increasing dislocation density.

§ 3. EXPERIMENTAL DETAILS

Most of the experiments were done on fine-grained, recrystallized Armco iron wires (3000 grains/mm²) in which large yield points can be developed. Subsidiary tests were made on coarse-grained Armco iron and on both coarse-grained and fine-grained high-purity iron†. The main impurities (in wt.-%) in the two irons were :

	C	Si	S	P	Mn	Ni	O ₂	N ₂
Armco	0.03	0.005	0.03	0.007	0.08	0.03	—	—
High purity	0.0068	0.002	0.005	0.001	0.0009	0.0024	0.003	0.001

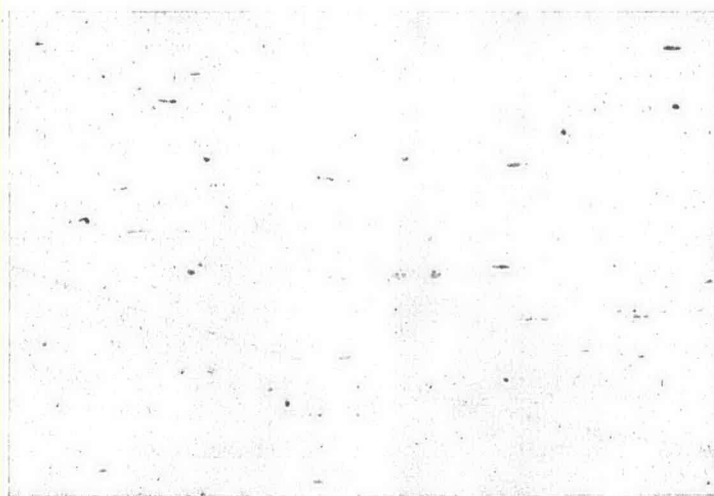
Wires were prepared by swaging and drawing to 0.048 in. diameter with intermediate annealing treatments. The grain size, which was measured by an A.S.T.M. graticule attachment, was varied by changing the stage of the penultimate anneal. Specimens were finally recrystallized over a short central length *in vacuo* at 700°C. 'Centre-annealing' eliminates the effect of stress concentrations at grips and enables both upper and lower yield stresses to be determined accurately (Hutchison 1957, 1963). Both irons contained non-metallic inclusions (fig. 1) ; but the density was considerably greater in Armco iron. No preferred orientation could be detected by x-ray examination.

Specimens to be pressurized were held freely in either kerosene or petroleum ether in a steel container ; load was applied slowly through a plunger, maintained for 5 min, and slowly released. Pressures up to 10⁴ atmospheres could be applied.

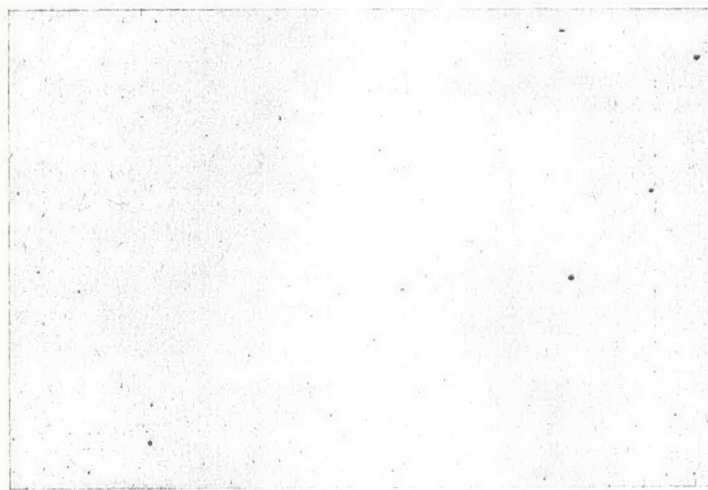
Pressurized specimens were either tested in tension immediately or stored in solid CO₂ until tested. Tensile tests were done in an Instron machine at a cross-head movement of 0.2 in./min ; load and elongation were recorded autographically.

† Kindly supplied by the British Iron and Steel Research Association as sample AJL16.

Fig. 1



(a)



(b)

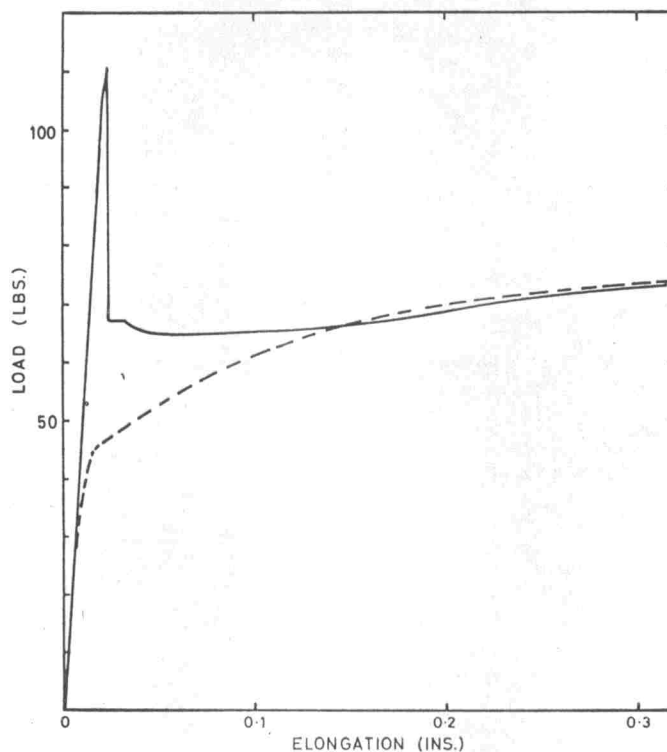
Mechanically polished surfaces of (a) Armco and (b) high-purity irons.
Unetched $\times 150$.

§ 4. EXPERIMENTAL OBSERVATIONS

4.1. Armco Iron

After pressurizing at 10^4 atmospheres, the yield point at atmospheric pressure and temperature is almost completely suppressed and plastic deformation occurs at stresses much below the previous lower yield stress (fig. 2). The absence of a yield point in pressurized material shows that free dislocations are present. Load-elongation curves of both unpressurized and pressurized wires coincide beyond the yield (fig. 2) indicating that the friction stress (Petch 1953) and the mechanism of deformation are unaffected by pressurization. No detectable plastic deformation occurred during pressurization; macroscopic dimensions were unchanged and no lattice disorientation or residual stresses were found by x-ray examination.

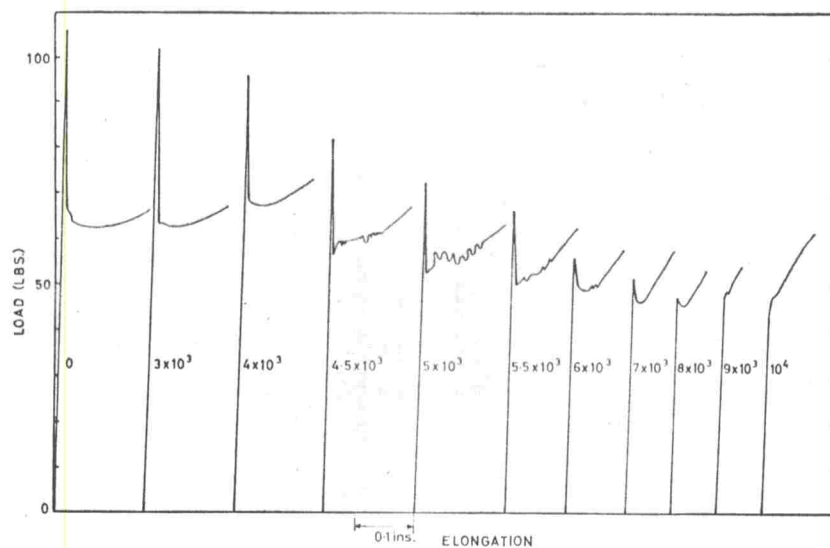
Fig. 2



Load/elongation curves at atmospheric pressure and temperature for Armco iron as recrystallized (solid curve) and after pressurizing at 10^4 atmospheres (dotted curve). (Note that the yield stress is reduced by more than half.)

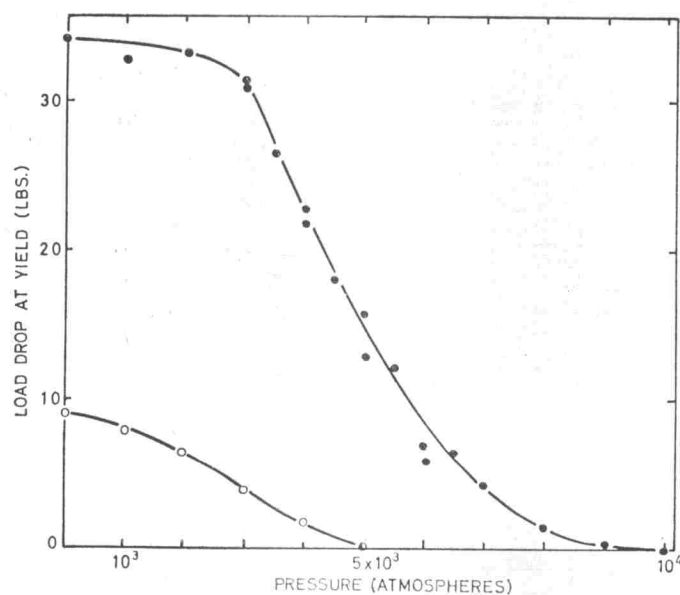
Specimens were subjected to different pressures and then tested at room pressure; fig. 3 shows the disappearance of the yield point with increasing pressure. Plotting the load drop at yield against pressure for different grain diameters shows that the yield disappears rapidly beyond

Fig. 3



Load/elongation curves at atmospheric pressure for Armco iron specimens subjected to the pressures shown.

Fig. 4

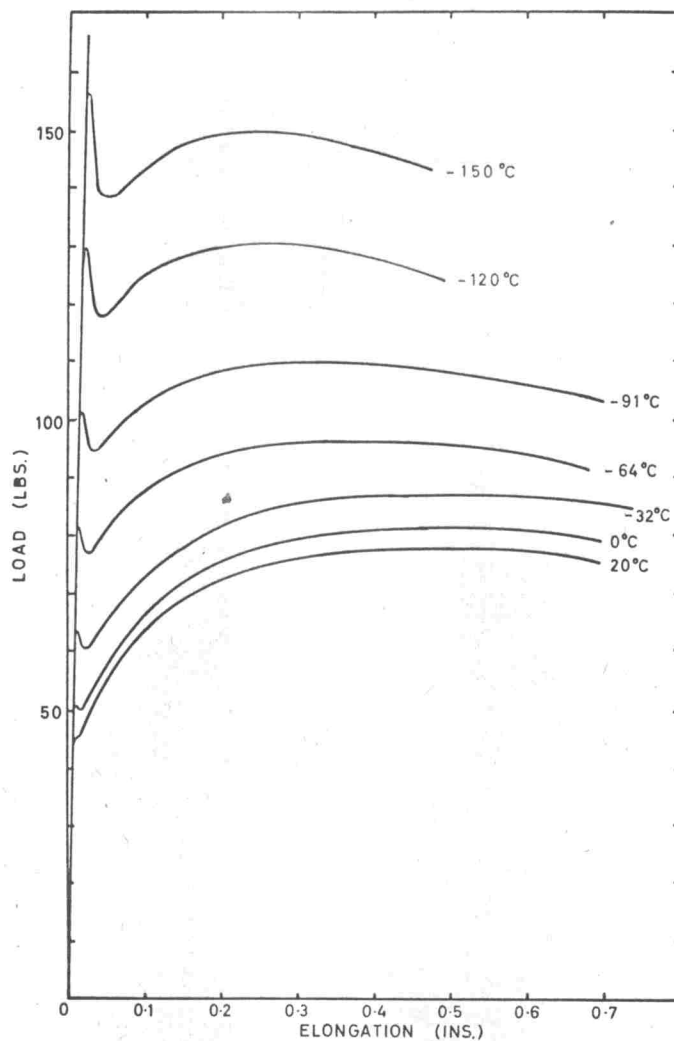


The load drop on yielding at atmospheric pressure plotted against the degree of pressurization (from the curves of fig. 3). Armco iron specimens. The upper curve is for a grain size of 3000 grains/mm² and the lower for 60 grains/mm².

some fairly critical pressure (fig. 4); the yield disappears at lower pressures in the coarser-grained specimens.

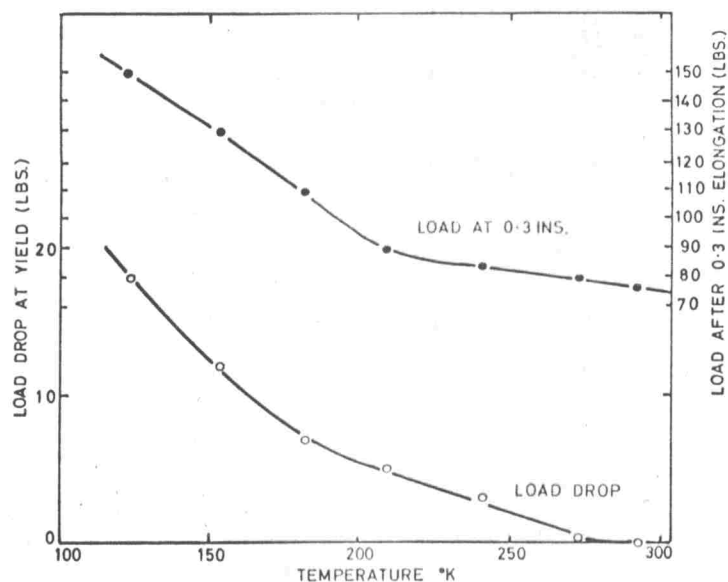
Load drops occur when pressurized specimens yield at low temperatures (fig. 5); these yields have a characteristic rounded shape, quite different from the yield in recrystallized material. The magnitude of the load drop varies markedly with temperature, in contrast to that in recrystallized iron which is almost independent of temperature (Hutchison 1963). The flow stresses at the various temperatures are the same for both recrystallized and pressurized specimens. The load drop at yield in pressurized

Fig. 5



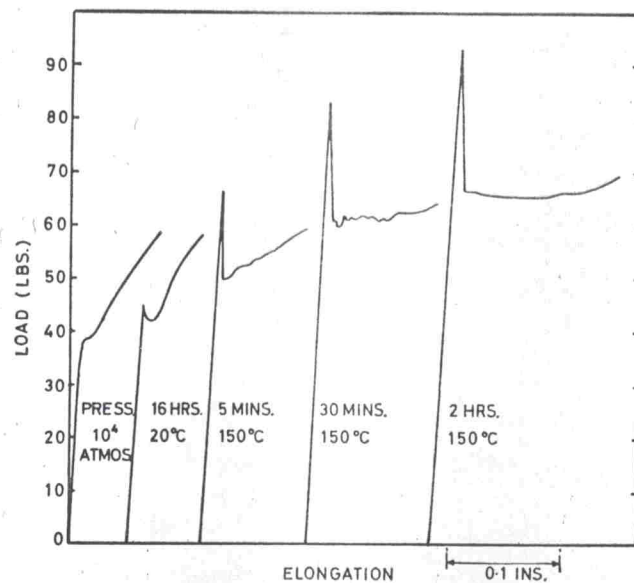
Load/elongation curves at different temperatures for Armco iron pressurized at 10^4 atmospheres.

Fig. 6



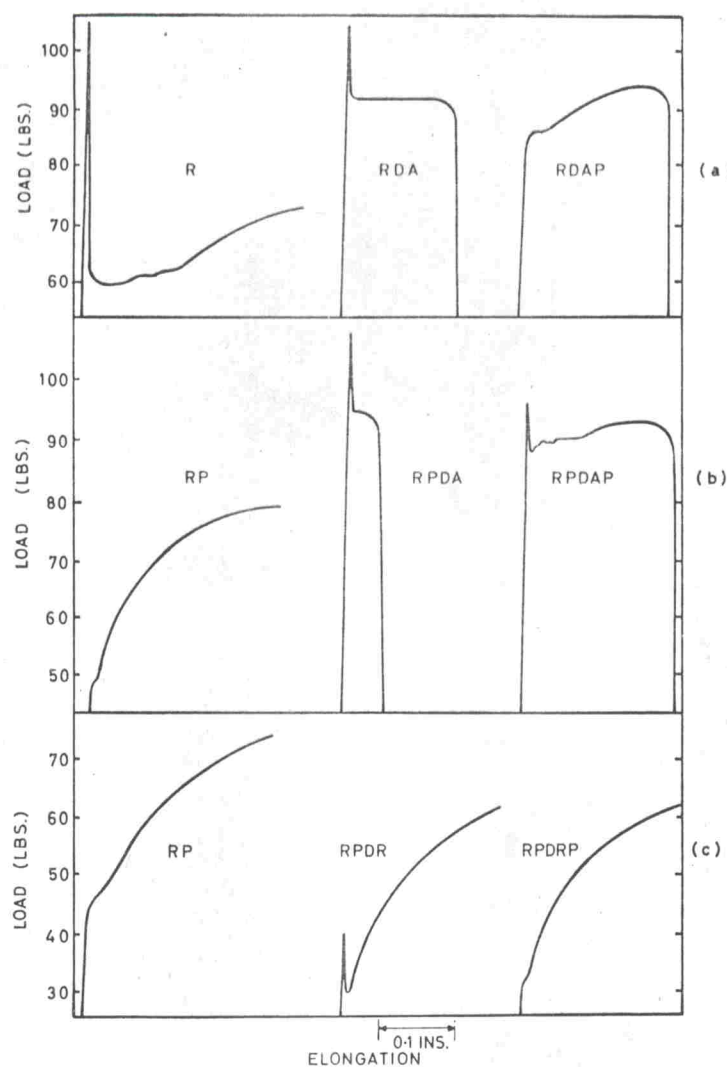
The load drop at yield and the flow stress after an elongation of 0.3 in. as a function of temperature for Armeo iron pressurized at 10^4 atmospheres.

Fig. 7



Load/elongation curves for specimens aged as shown after pressurization at 10^4 atmospheres.

Fig. 8



The effect of repeated pressurization. The letters on the diagrams refer to the condition of the specimen at the start of each test according to the following code:

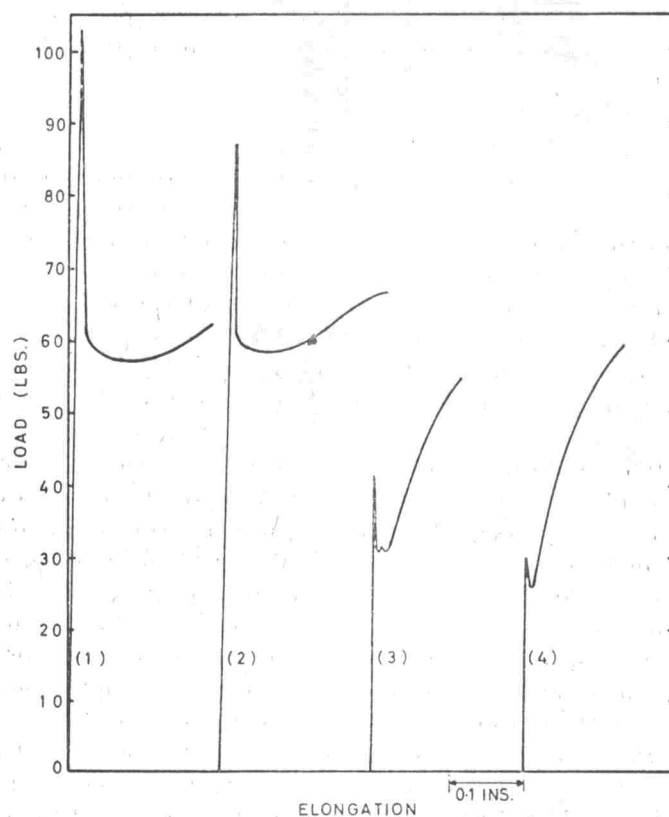
R: recrystallized at 700°C; P: pressurized at 10^4 atmospheres; D: elongated by 0.3 in.; A: aged at 150°C for 2 hr.

(a) shows that pressurization removes yield point in strain-aged specimens; (b) shows that pressurization has a much smaller effect in specimens which had been pressurized before strain-ageing; (c) shows that pressurization removes the yield point in specimens which were recrystallized after an initial pressurization.

specimens and the flow stress at given strain both increase rapidly with decreasing temperature below about 200°K (fig. 6). The transition in flow stress occurs because of the marked temperature-dependence of the friction stress below 200°K (Heslop and Petch 1956, Hutchison 1963). However, the friction stress is not a component of the load drop, although it is a component of flow stress. The significance of this observation will be considered later.

Figure 7 shows that the yield point at room temperature returns when pressurized iron is aged at temperatures where solute can diffuse to lock dislocations. This effect has been used to determine the influence of repeated pressurization; it has been found that after pressurization and ageing (at 150°C) a further pressurization has little effect on the yield. Similar tests have been done in which specimens have been deformed equal amounts, aged at the same temperature, and then pressurized.

Fig. 9



Load/elongation curves at atmospheric pressure and temperature for high purity iron as recrystallized (curves 1 and 3) and after pressurizing at 10^4 atmospheres (curves 2 and 4). Curves 1 and 2 refer to specimens of 1500 grains/mm² and 3 and 4 refer to 15 grains/mm².

Pressurization after strain ageing has little effect on specimens which were pressurized before the initial deformation (fig. 8 *b*), whereas the yield after strain-ageing is almost completely removed in specimens not initially pressurized (fig. 8 *a*). Further specimens, initially as-recrystallized and as-recrystallized and pressurized, were deformed by the same amount and then annealed at a temperature (700°C) sufficient to cause recrystallization; subsequent pressurization removed the yields in both cases (fig. 8 *c*). We conclude that the effect of pressurization is exhausted by a single application of pressure unless recrystallization occurs between pressurizations.

4.2. High-purity Iron

Figure 9 shows that pressurization has little effect on the yield point in specimens of similar grain sizes to the Armco iron. As in the case of Armco iron, the effect is greater in the coarser-grained specimens. However, the yield point in coarse-grained pure iron is only slightly reduced after pressurization at 10^4 atmospheres, whereas pressures of 5×10^3 atmospheres completely removed the yield point in the coarser-grained Armco iron.

§ 5. DISCUSSION

The disappearance of the yield point leaves little doubt that free dislocations are formed in Armco iron during pressurization; the question at issue is their origin. Free dislocations could result if pressure (*a*) causes plastic deformation, thereby removing existing dislocations from their locking atmospheres or creating new dislocations, or (*b*) irreversibly affects the solute atmospheres around dislocations, e.g. by promoting local precipitation or lattice transformation. The experimental observations are consistent with plastic deformation during pressurization.

Hahn (1962) and Cottrell (1963) have considered the effect of free dislocations on plastic yielding; their predictions closely match the present observations. The gradual disappearance of the yield point with increasing pressure (fig. 3) is consistent with the effect of an increasing density of free dislocations. The rounding of the yield before its disappearance is predicted by Hahn and Cottrell; under simultaneous multiplication of dislocations and strain-hardening, the stress/strain relationship at yield is a continuous function. The similar temperature-dependences of the friction stress and the load drop at yield in pressurized specimens (fig. 6) are also expected because the temperature-dependence of the stress for a given dislocation velocity matches that of the friction stress (Stein and Low 1960). The applied strain rate and the density of free dislocations are the same at different temperatures, so that the same dislocation velocities are required; the maximum load at yield follows the driving stress required for this velocity and hence has the same temperature dependence as the friction stress.

Considerable evidence exists for pressure-induced lattice transformations, but pressures far beyond 10^4 atmospheres are usually required in iron (Kaufman *et al.* 1960). The present observations disagree with an explanation on this basis. For instance, specimens deformed equally and aged at the same temperature differ in their response to pressurization according to whether or not they were pressurized before deformation (fig. 8); the atmospheres around dislocations should not differ appreciably, so that similar transformations would be expected in both cases. Similarly, the effect of pressure on solute atmospheres should not depend markedly on grain diameter; in fact the yield disappears at lower pressures in coarser-grained specimens (fig. 4). Finally, the temperature-dependent load drops in pressurized iron (fig. 6) could arise from weak interaction between dislocations and precipitated or transformed regions, but why should the temperature-dependence match that of the friction stress?

The evidence in favour of plastic deformation during pressurization is far more convincing. Even the failure to detect the deformation in pressurized specimens is not surprising in view of the sensitivity of the yield point to low densities of free dislocations. Hahn (1962) estimates that the yield point will be considerably reduced by $\sim 10^3$ dislocations/cm². Our experiments show that just sufficient dislocations are generated to remove the yield point at room temperature; the macroscopic deformation corresponding to the formation of this density of dislocations would not be detected by normal methods.

Hydrostatic pressure can only cause plastic deformation at elastic inhomogeneities, such as particles of second phase or voids; the linear compressibility of iron is isotropic (Bridgman 1949) so that grains contract equally whatever their orientation. The formation of dislocations around particles under hydrostatic pressure should be strictly analogous to that occurring around particles during cooling (Jones and Mitchell 1958). Iron undergoes profuse cross-slip, so that particles are likely to retain 'dislocation atmospheres' when the pressure is released. The stresses from these atmospheres would tend to prevent the formation of new dislocations on repressurizing; this would explain why the yields are not affected by pressurization in strain-aged specimens which were initially pressurized (fig. 8*b*). The collapse of voids during the initial pressurization would explain the failure of aged specimens to respond to repressurization, but it is unlikely that voids would re-open during recrystallization at 700°C; fig. 8(*c*) shows that specimens do respond if recrystallized between pressurizations. It is quite reasonable, however, to expect that the 'dislocation atmospheres' around particles would disappear during recrystallization; a further pressurization would then be fully effective. The small influence of pressurization on the yield point in high-purity iron (fig. 9) is also consistent with this interpretation, because the high-purity iron contains very few particles compared to the Armco iron (fig. 1).

The variation of the effect of pressure with grain size (fig. 4) is surprising since the dispersion of particles is unlikely to vary, particularly as all specimens were recrystallized at the same temperature. However, the different effects of pressure on the yield points do not necessarily mean that different numbers of dislocations are generated during pressurization. The estimate that $\sim 10^3$ free dislocations/cm² are formed during pressurization suggests a similar (or lower) density of active particles. If the particles are randomly dispersed, the *proportion* of grains containing active particles will vary as (grain diameter)³. Since grain boundaries offer by far the greatest resistance to the spread of deformation from prematurely-yielded regions, the effectiveness of each active particle will depend on the volume into which its dislocations can spread and multiply freely, i.e. on the grain volume. In fine-grained material, for instance, the deformation will be confined to small volumes around particles, and the few particles which are active may not significantly increase the usual number of prematurely yielded grains ahead of the Lüders band (Low 1961). In coarse-grained material a similar density of active particles may initiate deformation within most grains, without deformation having to propagate across a grain boundary. Hence, the same density of active particles will have vastly different effects on yielding, depending on the number of grains containing active particles, i.e. on the grain size.

§ 6. CONCLUSION

Pressurization at several thousand atmospheres markedly affects the yield point at atmospheric pressure. Evidence has been advanced to show that the effect arises because of the different compressibility of particles of second phase; under hydrostatic pressure, this causes plastic deformation and the formation of free dislocations. The effect can only be detected because of the extreme sensitivity of the yield point to free dislocations.

The behaviour of pressurized iron is qualitatively in agreement with recent theories of yielding in materials containing free dislocations (Hahn 1962, Cottrell 1963).

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