

◁Original▷ **The Effect of Compression on Strain
Ageing of Ferrovac E Iron**

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Abstract

The effect of compression in the strain ageing of Ferrovac E iron has been examined with compressive testing following the prestrain in compression. Both prestraining and testing were performed at room temperature with the strain rate of $1.9 \times 10^{-4} \text{ sec}^{-1}$ and that of $0.95 \times 10^{-4} \text{ sec}^{-1}$, respectively. Ageing was carried out at several temperatures below 80°C using thermostatically controlled oil baths with a temperature control within $\pm 1^\circ\text{C}$.

It was found that the rate of strain ageing obeyed the $t^{2/3}$ law up to about five hours ageing at 60°C . The rate was slower than those⁸⁾ reported in case of the tensile prestrained iron. Activation energy for strain ageing has been estimated as 21.5 Kcal/mole at the first stage of the aging process. At the second stage of ageing where the $t^{2/3}$ law is still valid, however, the activation energy was somewhat decreased. The activation energy at the first stage of ageing was about 10% larger than published results on the tensile-prestraining. This difference between the activation energies is explained in terms of the residual stress field in lattice.

요 약

압축변형시킨 순철(0.007% 탄소포함)을 80°C 이하에서 저온열처리(ageing)한 후, 열처리시간에 따라 증가하는 저항복점(lower yield point)의 변화를 압축시험으로 측정하여 그 strain ageing 효과를 조사했다.

본 실험에서 압축변형된 순철의 strain ageing 속도는 저항복응력의 증가가 그 최고 증가의 60% 정도에 이를 때까지 열처리시간의 2/3승에 비례했으나, 이미 알려진 인장변형의 경우에서보다 느렸다. 이 60%의 증가는 순철을 60°C 에서 약 5시간 열처리함으로써 얻었다.

압축변형된 순철의 strain ageing 을 위한 활성화에너지는 열처리 초기단계(60°C에서 약 30분)에서 21,500 cal/mole 이었는데, 이것은 인장변형의 경우에서 알려진 것보다 대략 10%가 큰 값이다. 이 증가는 잔류응력에 의해 결정내에 형성되는 strain field 로 설명되었다. 열처리공정의 둘째단계(60°C에서 약 5시간까지 계속되는)에서는 그 활성화에너지가 다소 감소되는 경향이 있었다.

1. Introduction

In some metals and alloys, the discontinuous yield points reappear when the material has been subjected to a low-temperature heat treatment(aging) following prestraining. This is the phenomenon of strain ageing.

This yield phenomenon is particularly marked in mild steel and soft iron containing small amounts of carbon and nitrogen. It is well established that strain ageing of α -iron is due to the diffusion of the interstitial solute atoms such as carbon and nitrogen into free dislocations which are generated during plastic deformation¹⁻⁷.

If a specimen is loaded beyond its yield point (Lüders strain) and subsequently unloaded, it contains mostly free dislocations and will not show a discontinuous yield point on immediate reloading. However, if it is aged prior to reloading, the solute atoms have time to migrate to the dislocations to form new Cottrell atmospheres which anchors the dislocations strongly. Only when the applied stress is large enough to overcome this anchoring force, the anchored dislocations can break away and move freely through the lattice. Once the dislocations are free in this way, then the stress necessary to keep them in motion is lower than the stress required for the dislocations to break away the atmospheres.

Cottrell and Bilby showed that the number of solute atoms per unit volume of α -iron migrating to dislocations is proportional to the $2/3$ power of ageing time, t and the solute concentration initially present in the material. This Cottrell and Bilby's theory representing

the stress induced diffusion gives good agreements with experimental data on the first stages of ageing. Harper²), however, modified the Cottrell-Bilby theory for the later stages of ageing by saying that the migration rate of solute atoms should be proportional to the concentration left in solution at any instant. And recently Humphreys *et al.*⁹) and Hartely¹⁰) have shown that the migration rate at a given temperature is proportional to $t^{1/2}$ instead of $t^{2/3}$.

The solute segregations to dislocation continues even after all the strong interacting dislocation sites are occupied by one or two solute atoms per atomic plane^{6, 8}).

There have been several measurements^{2, 3, 4, 11}) on the activation energy for the stress-induced diffusion of carbon atoms in α -iron. The observed values of the activation energy for the stress-induced diffusion in α -iron are generally somewhat larger than that for the concentration gradient-induced diffusion in α -iron, although most investigators have assumed both values to be the same magnitude^{2, 3, 12}).

Humphreys *et al.*⁹), however, suggested that the activation energy for diffusion of a particular carbon atom to a favorable site depends inversely on its distance from the dislocation line. And he pointed out that the amount of energy required to surmount the activation energy barrier which must come from an external source is lowered because some of the required energy has already been supplied in the form of lattice strain energy. But it is thought that Humphreys seemed to disregard the possibility of difficulty in diffu-

sion of carbon atoms to dislocation owing to the lattice distortion around the dislocations.

Most of the strain ageing effects have been studied with the application of tensile force on mild steels or pure iron which have been prestrained in tension and aged. Polakowski¹³⁾ and Tardiff¹⁴⁾ suggested that the method of prestraining do not affect the basic mechanism of the strain ageing, but that the cold working invoking inhomogeneous residual stress retard the strain-ageing effect. As yet satisfactory explanations have not been offered for the reason why the inhomogeneous residual stress retards the return of the discontinuous yield point. And the strain ageing effects by compression have not been reported, too.

The purpose of this investigation is to examine the effects of compression for the strain ageing with an application of compressive force on an aged pure iron which has been subjected to compressive prestraining.

The deformation mechanisms in tension and in compression are basically the same except in directions of slip and applied forces. In compressive deformation, the compressive normal force to the slip plane will suppress the crystal. As a result, the interplanar spacing of the slip plane will become somewhat shorter in compression, while that in tensile deformation may be elongated.

If a part of the residual stress introduced during plastic deformation is assumed to be present in the form of lattice distortion, it can be expected that there will be a remnant of some decrease in the interplanar distance across slip plane even after unloading in case of compressive deformation. Thus the suppression of the crystal lattice would make the solute diffusion during ageing treatment of the precompressed pure iron difficult and somewhat raise the energy barrier of activation for strain ageing, as compared with the case of tensile deformation.

2. Experimental Procedure

(1) Materials and Specimen Preparation

The material used was a Ferrovac E iron rod with a purity of 99.9% containing 0.0073% interstitial solutes (0.007% C and 0.0003% N).

The iron rod with a diameter of 6.35 mm was cut into cylindrical specimens having dimensions as specified in Fig. 1(a). Then the cut face was mechanically polished with emery papers starting with No. 2 and finishing with No. 5.

After polishing, all the specimens were vacuum-annealed and solution-treated for 30 minutes at 700°C in a tube furnace and then quenched in water at the room temperature. The stainless steel combustion tube was evacuated to a vacuum of about 10^{-3} mmHg and the vacuum was kept on during the heat treatment.

Microscopic examination showed that the heat-treated specimen had an average grain size of about ASTM 7.5, and that the elongated grain of the as-received material was considerably modified to equiaxed grain.

(2) Testing Procedures

The thin oxide film produced on the specimen surface during the heat treatment was removed by a light polishing using 500G silicon caricide paper.

Special cautions were introduced to the specimen preparation and the polishing. It has been shown that even a very small tilt angle between the loading axis and the normal to the end face of a specimen results in appreciable variations of data in compression test¹⁴⁾. Preliminary test showed that the preparation technique of specimen was satisfactory for the present experiment.

The heat-treated and polished specimens were all prestrained to the 5% compressive strain prior to ageing. This 5% reduction amounted to the end of the Lüders strain of

the annealed and quenched specimens. Immediate reloading without ageing, therefore, will show no discontinuous yield point phenomena.

The precompressed specimen had a convexed cylindrical shape as shown in Fig. 1(b). Thus the calculated average value was used as the cross-section area of the specimen.

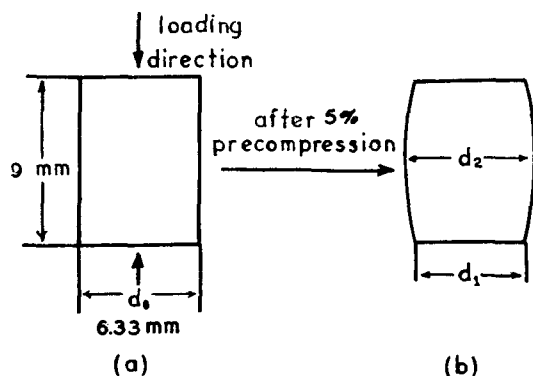


Fig. 1. Dimension of the initial specimen(a) and the shape of the precompressed specimen(b).

The prestrained specimens except those used for the activation energy measurements were all aged in thermostatically controlled oil baths of 60°C with a temperature control within $\pm 1^\circ\text{C}$.

The experimental variables determining activation energy were times and temperatures. However, the experimental conditions in this study were standardized so that only one experimental variable was the ageing temperature. For the activation energy data, ageing treatment was performed for 30 minutes at several temperatures, *i.e.* 44, 47, 51, 60, 67, 70, and 80°C.

Immediately after ageing, all the specimens were compression-tested using a hard beam universal testing machine, Shimadzu Autograph.

Both precompression and test compression were carried out at room temperature at the strain rate of $1.9 \times 10^{-4} \text{ sec}^{-1}$ and that of $0.95 \times 10^{-4} \text{ sec}^{-1}$, respectively.

3. Results

(1) General Results

Of the complete series compression-tested after 5% precompression and ageing at 60°C, the representative true stress-strain curves are shown in Fig. 2. They illustrate the main features of a sequence of changes due to strain-ageing. The ageing process may be divided into the following stages:

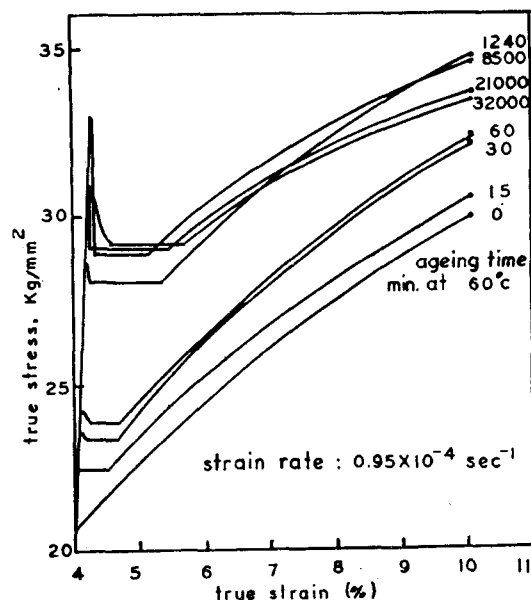


Fig. 2. Progressive changes in the compressive stress-strain of Ferrovac E iron due to ageing at 60°C after 5% precompression.

(a) During the first 30 min. of ageing at 60°C the lower yield stress increased rapidly. At the end of this stage the upper yield point began to appear.

(b) Beyond the ageing of about 30 min., both the lower yield stress and Lüders strain showed an increase but rather slower than those at the first stage of ageing. Up to the ageing of about 1,240 min., the upper yield stress increased slowly.

(c) On ageing for more than about 1,240 min., the lower yield stress continued to increase but very slowly. At the ageing of 8,500

min., the maximum upper yield stress was detected but it decreased appreciably on further ageing. At this stage some evidence of decrease in the Lüders strain was detected.

For the study of the kinetics of strain ageing, several parameters have been suggested, *i. e.* — the changes in the upper yield stress over the yield stress before ageing¹⁵⁾, the changes in hardness¹⁶⁾, the changes in Lüders strain^{4, 6, 8)} and the changes in electrical resistivity^{13, 17)}. Selections of these parameters have been based on the fact that they are proportional to the amount of solutes migrated to dislocations during the early stage of ageing, which obeys the $t^{2/3}$ law of Cottrell and Bilby.

Wilson⁸⁾ suggested that the change in lower yield stress is closely proportional to the number of solute atoms locked in dislocations up to the second stage of ageing. For the present purpose, thus the fractional change in lower yield stress f was selected as follows:

$$f = \frac{\Delta\sigma}{\Delta\sigma_{max}}, \quad (1)$$

where $\Delta\sigma$ is the increase in the lower yield stress after ageing and $\Delta\sigma_{max}$ the maximum increase in the lower yield stress after full ageing.

(2) Relation of Changes in the Lower Yield Stress to Ageing Time

The lower yield stress was measured from each curve in Fig. 2. The results were plotted in Fig. 3. as a function of ageing time at 60°C. The σ_o was obtained by reloading the quenched specimen immediately after precompression. An average value of the minimum lower yield stress σ_o , corresponding to $\Delta\sigma=0$, was 21.8 kg/mm². σ_{max} was obtained by extrapolation at the end of the complete ageing (about 4×10^4 min). σ_{max} , the maximum value of the lower yield stress, was calculated to be about 29.3 kg/mm.

To gain a quantitative picture of the rela-

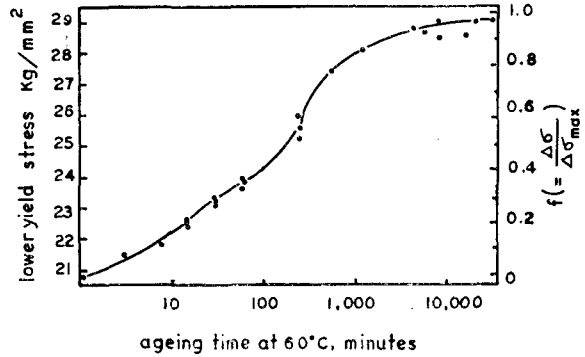


Fig. 3. Relationship between changes in lower yield stress of a Ferrovac E iron and ageing time after 5% precompression.

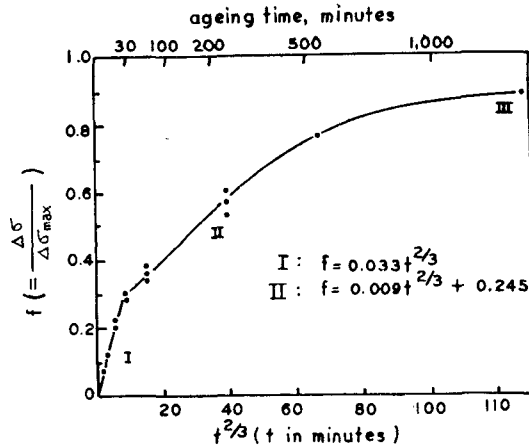


Fig. 4. Fractional changes (f) in the compressive lower yield stress as a function of $t^{2/3}$ for a Ferrovac E iron aged at 60°C after 5% precompression.

tionship between f and ageing time, f was plotted as a function of $t^{2/3}$ in Fig. 4. Fig. 4 shows that f obeys a $t^{2/3}$ law up to about 60% completion of the maximum measured change. This linear relationship can be divided into two parts; say, regions I and II as shown in Fig. 4.

The first stage of ageing depicted by the region I was completed after about 30 min. ageing and at this stage $\Delta\sigma$ increased up to about 30% $\Delta\sigma_{max}$. At the second stage of ageing (region II), the increase in f became slower. Nevertheless, its linear relationship with $t^{2/3}$ still kept on. This second stage

continued up to an ageing time of roughly 300 min. In this case, the increase in the lower yield stress reached about 60% of its maximum value around its point of departure from the linear relationship.

As shown in Fig. 4, the fractional changes in the lower yield stress of specimens aged at 60°C is a function of ageing time:

$$f = \frac{\Delta\sigma}{\Delta\sigma_{max}} = Mt^{2/3} + N \quad (2)$$

where M is the slope of the linear curve and N the intercept of the curve with the fractional changes in lower yield stress axis. In the first stage of ageing (region I), M had a value of 0.033 and N was zero. In the second stage M was 0.009 and N 0.245.

Changes in the lower yield stress of specimens aged for 30 min. at several temperatures were plotted in Fig. 5 as a function of ageing temperature.

Using eq. (2) and the changes in lower yield stress due to ageing temperature in Fig. 5, each ageing temperature was calculated to the corresponding time if ageing is performed at 60°C. The corresponding ageing times were plotted on the upper axis in Fig. 5.

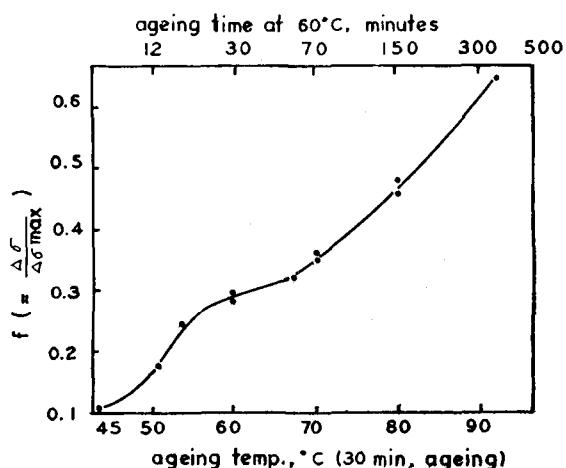


Fig. 5. Changes in the compressive lower yield stress f of Ferrovac E iron with ageing temperature and with the calculated ageing time corresponding to that at 60°C.

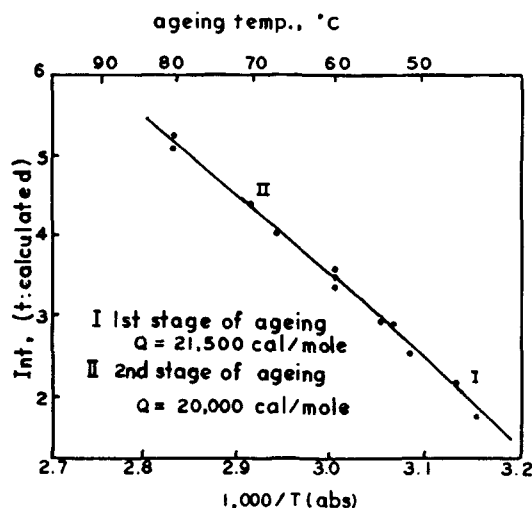


Fig. 6. Activation energy for strain ageing of a Ferrovac E iron after 5% precompression.

Because the ageing process is controlled by the thermally activated rate process, the relationship between the ageing temperature and the corresponding ageing time would follow Arrhenius equation:

$$t \propto A \exp(-Q/RT) \quad (3)$$

Where A is a constant, R the gas constant, Q activation energy for diffusion, T absolute temperature, and t time for the process to reach an arbitrary stage.

Thus the logarithms of the corresponding time were plotted against $1/T$ in Fig. 6. From the slope of the curve, the activation energy for first stage of ageing was estimated to be 21.5 Kcal/mole using least mean square method. In the second stage the activation energy was 20 Kcal/mole.

4. Discussions of Results

From the results, the first stage of ageing was finished at about 40 min. The ending time of the second stage was roughly 300min.

The amount of solute atoms per unit volume of α -iron migrated to dislocations at ageing temperature T after ageing time t can be calculated from the Cottrell-Bilby equation¹⁾:

$$q=n(t)=3\left(\frac{\pi}{2}\right)^{1/2}n_0L\left(\frac{ADT}{kT}\right)^{2/3} \quad (4)$$

where n_0 is the solute concentration in solution at zero ageing time, L the dislocation density, A the interaction parameter of solute atoms with dislocations, and D the diffusion coefficient of the solute in α -iron. The value of A has been estimated in the range 1.4 to 3×10^{-20} dyn cm² for carbon and nitrogen in iron¹¹. The Cottrell-Bilby equation is known to be valid at the early stage of ageing.

Diffusion coefficient D at a certain temperature can be calculated from the equation,

$$D=D_0 \exp\left(-\frac{Q}{RT}\right) \quad (5)$$

Here D_0 is a constant which has the value of 0.0073 cm²/sec. and Q is the activation energy for the diffusion having the value of $18,100$ cal/mole for carbon in-iron¹⁸.

It is reasonable to assume, from the result of other investigators^{2, 8}, that the dislocation density L is 10^{11} /cm² for this moderately deformed and aged Ferrovac E iron containing 0.0073% (C+N).

In this way, the q value in eq.(4) can be obtained by putting $A=2 \times 10^{-20}$ dyn cm². As a result of this, about 8 per cent of the total solute contents can be known to migrate to the extended regions along the dislocations during the ageing of 30 min. at 60°C. This migrated solute corresponds to the amount if the solute atoms occupy the interaction sites along the dislocations at intervals of about 2.5 cm⁻⁸. This distance 2.5 cm⁻⁸. agrees well with the mean atomic spacing along the dislocations. The mean atomic spacing was assumed to be twice the radius (1.24 Å) of iron in this estimation. This means that all the dislocations have been locked by one solute atom per atom plane at the end (30 min. at 60°C) of the first stage of ageing. In other words, all the strong interaction sites of solute atoms with dislocations become completely

occupied at the end of this ageing stage. Therefore, 0.006% (C+N) in the Ferrovac E iron would be sufficient for the complete atmosphere locking. This first stage of ageing thus defined by the complete locking is depicted by the region I in Fig. 4.

Segregation of solute to dislocations does not stop even after the first stage of ageing because the stress field around dislocation is not relieved completely by the complete locking^{11, 4, 6}. But the subsequent segregation has little or no effect on the stress required to unpin the locked dislocations⁶, since all the strong interaction sites have been already occupied. This second stage of ageing depicted by region II (Fig. 4) can be defined clearly, for the effectiveness of the solute in raising the yield stress changes rather suddenly immediately after the complete locking. Further rises in the yield stress at this stage are due almost entirely to changes in microstructure which increase resistance to the movement of mobile dislocations⁸. At the latest stage of ageing beyond the region II, the amount of solutes available for the ageing process will be considerably reduced. This is considered to cause an immediate reduction in increases in the yield stress.

As shown in Table 1, Wilson⁸ reported that the ageing time for complete locking is 4 min. at 60°C for a tensile-deformed iron containing 0.014% (C+N) and 33 min. for 0.0022% N.

In case of 0.0022% of Wilson's specimens, the solute contents is much less than that of the present work. But the ageing times for complete locking are much the same. And it is well known that the diffusion of nitrogen in α -iron is easier than for carbon. Thus the ageing time of 30 min. in the present experiment can be known to be rather long compared with tensile-deformed iron.

Table 1. Comparison of the rate of strain ageing between in the present result and in the published tensile-predeformed data

	Wilson, <i>et al.</i> ⁹⁾		Present work
Available solute (wt. %)	0.01C+0.004N	0.0022N	0.007C+0.0003N
Prestrain	tension 4%	tension 4%	compression 5%
Dislocation density (lines/cm ²)	1.0×10^{-11}	5.1×10^{10}	1.0×10^{11}
Test method	tension	tension	compression
Atosphere density at complete locking (atoms/atom plane)	1.2	1.1	1.0
Ageing time for complete locking at 60°C (min.)	4.0	33	30

Table 2. Reported activation energies for the strain ageing of α -iron

Solute	Prestraining method	Test method	Activation energy, Kcal/mole	Reference
C	Tension	Tension	18.8	Tardif ³⁾
	Temper rolling	Tension	19.7	Tardif ⁴⁾
	Without	Internal friction	19.16	Lord <i>et al.</i> ¹¹⁾
	Tension	Internal friction	20	Harper ²⁾
	Tension	Internal friction	20.4	Thomas ³⁾
N	Tension	Internal friction	17.2	Harper ²⁾
	Tension	Internal friction	18	Thomas ³⁾
C+N	Compression	Compression	21.5	Present work

In the light of the above discussion, it would be suggested that rate of strain ageing is somewhat slower in α -iron for the case of compression than in tension. This fact can be interpreted from the change of activation energy for strain ageing.

In the present experiment, the activation energy for the strain ageing of the precompressed Ferrovac E iron was 21.5 Kcal/mole at the first stage of ageing. The published results for activation energies are shown in Table 2.

It has been known that the activation energy for the diffusion in α -iron is 18.1 Kcal/mole¹⁸⁾ for carbon and 16.4 Kcal/mole¹⁹⁾ for nitrogen.

Nabarro¹²⁾ analyzed other published data and showed that the activation energy for strain ageing process is of similar magnitude with that for the diffusion of the solutes in α -iron. The data in Table 2, however, show that the activation energies for strain ageing are somewhat larger than that for the diffusion.

It can be seen also from the data that activation energy measured by internal friction method is generally larger than that by mechanical test.

In consideration of these, some conclusions may be followed: Prior deformation will increase the activation energy for diffusion of the solutes in α -iron. The activation energy may become larger for the prestraining by compression including rolling than by tension.

This increase in activation energy in the first case would be possibly explained in terms of the lattice distortion around dislocations. When a metal undergoes a plastic deformation, about 10% of the input energy is known to be stored to increase dislocation density and other defects. As a result of this, the strain energy around dislocations may be increased owing to the shortening in the inter-dislocation distance. This increased lattice strain energy will lower the amount of energy required to surmount the activation energy barrier⁹⁾. However, it cannot

be neglected that the elastically-distorted lattice due to input energy may make the carbon diffusion in α -iron difficult, and that it will raise the activation energy barrier which carbon atoms must overcome for diffusion. Within the limit of this experimental evidences, the quantitative comparison is not possible between the increase in activation energy barrier and the decrease in the activation energy due to plastic deformation. The present result, however, indicates that the former may be somewhat larger than the latter.

The differences in the values of activation energy between both in tensile deformation and in compressive deformation may be thought to be possibly due to the difference in the deformation model shown in Fig. 7. When a uniaxial tensile loading is applied to a specimen, lattices at the both sides of slip plane will receive tension force F_{tn} normal to the slip plane as shown in Fig. 7(a). On the other hand, compressive force F_{cn} normal to the slip plane will be exerted on the slip plane in case of compressive loading as shown in Fig. 7(b).

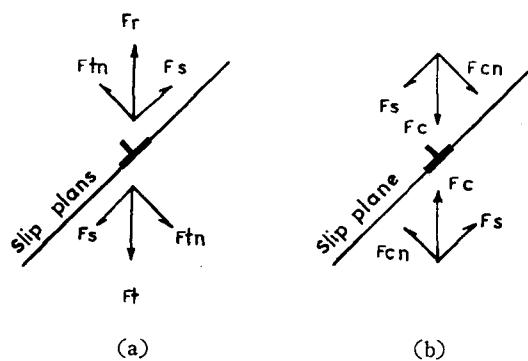


Fig. 7. Deformation model around dislocation in tensile (a) and compressive (b) loading of specimen.

When the specimen is unloaded after plastic deformation, most of the residual stress will remain as the form of defects (dislocations, vacancies and so on). It may not be wrong to suppose, however, a small part of the residual stress would be present in the form of com-

pression or tension in the lattice around dislocations near slip planes. This residual stress may suppress the lattice elastically in case of compression and it might elongate the lattice in case of tension. In this way, even if it were not of the form of lattice suppression, the small amount of the residual stress will cause to increase the internal friction relatively more in the case of compressive deformation. The increased internal friction will make the diffusion of the solutes to dislocations difficult. Thus the increase in the activation energy for strain ageing will be relatively larger in case of prestraining in compression than in tension. This will also retard the rate of strain ageing comparatively in case of compressive deformation.

The decrease in the activation energy at the second stage of ageing may be due to the relaxation of the strain around dislocations. During the atmosphere formation (the first stage), all the strong interaction sites of dislocation will be filled by solute atoms^{6, 8)}. This may somewhat relax the stress field around dislocation center. In this way the solute diffusion would be thought to be comparatively easy.

Changes in lower and upper yield stresses of the precompressed Ferrovac E iron typically showed similar features to those of Wilson's^{6, 8)} pre-extended low carbon steel. Upper yield stress and Lüders strain showed different features from those of Wilson's specimen.

5. Conclusion

Within the scope of the present investigation on Ferrovac E iron containing 0.007% carbon and 0.003% nitrogen, it is concluded that:

(1) The prior deformation regardless of deformation method may generally increase the activation energy for diffusion of interstitial solutes in α -iron.

(2) The activation energy for strain ageing in precompressed iron seems to be somewhat

higher than that in the pretensioned iron. The activation energy for strain ageing of 5% precompressed Ferrovac E iron was 21.5Kcal/mole at the first stage of ageing and 20Kcal/mole at the second stage. This activation energy at the first stage was about 10% larger compared with published values for pretensioned specimens.

(3) The rate of strain ageing was somewhat slower in case of prestraining in compression than that in case of tensile prestraining. This is thought to be due to the increased activation energy barrier around dislocations.

(4) The changes in lower yield stress were similar to those⁶⁾ of pretensioned iron. However, the changes in upper yield stress and Lüders strain seemed to be different from those of pretensioned specimens.

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