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Recrystallization behavior of a low carbon steel wire

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Abstract
The primary recrystallization of a steel containing 0.05 (wt%) carbon is examined. The present material is produced by Trifisoud- Setif –Algeria and received as wires of 4, 3.18, 2.45mm diameters. The samples are subjected to annealing treatments at 480-520°C. The recrystallization temperature is determined and found to be lower for the wire with higher wiredrawing area reduction (τw). The two parameters (k and n) in Avrami law are calculated. A relationship between τw and recrystallized grain size (drec) is established. The recrystallization activation energy is estimated. The obtained results are compared with other material results.

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1. Introduction:

Material is produced as large rods of 6 mm diameter. These rods are then further processed, by means of wiredrawing process, in wires of different diameters to final product. To a large extent the mechanical and structural properties of a metal depend on the dislocation density, the grain size and grain orientation [1,2,3] .The elongated grains in deformed material, due to the wiredrawing, result in unstable structure. An annealing treatment is necessary since it allowed a return to a stable condition. The annealing processes involve the migration of internal grain boundaries within the deformed material and the production of a new equiaxed grain structure. This process is termed recrystallization. Both nucleation and growth events constitute recrystallization and occur at any time throughout the material. The primary recrystallization rate is strongly depending on the temperature and time of annealing.

The primary recrystallization activation energy is referred to the process as a whole kinetics i.e. nucleation and growth. Such activation energy is expected to vary not only with the change of material composition but also with the amount of strain produced by mechanical processing [4]. It has already shown that the higher cold working, the lower is the recrystallization activation energy in CP-titanium [5].

The final recrystallized grain size is affected by both the initial grain size and the amount of deformation [6].

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Ferrite grain refinement, obtained after complete recrystallization, is an important objective of low carbon steels; since it ensures a balance between high strength and good toughness.

The present study is devoted first to the examination of primary recrystallization and also to the determination of recrystallization activation energy and final grain size.

2. Experimental procedure

The investigated material is low carbon steel wire supplied by the “Trifisoud, Setif, Algerie”. The material is received in long rods with different diameters of 6.4, 3.18 and 2.45 mm. The chemical composition is shown in table (1) and the reduction of area ($\tau_w$) i.e. deformation rates of wires, is given in table (2).

Table 1. Chemical composition (weight %)

| C   | Mn | Si | S   | P   | Ni  | Cr | Cu |
|-----|----|----|-----|-----|-----|----|----|
| 0.05| 0.3| 0.1| 0.025| 0.02| 0.12| 0.12| 0.12|

Table 2. Deformation rates

| $\phi$ (mm) | 06 | 04 | 3.18 | 2.45 |
|-------------|----|----|------|------|
| $\tau_w$ (%)| 0  | 55.5 | 72  | 83 |

The material is formed using a wiredrawing machine BREITENBACH type Standard 1R/4VZ, the process is carried out at room temperature using a soap of silicate as a lubricant.

Optical metallographic examination, using a ZEISS microscope equipped with AXIO-VISION software used for image analysis, is carried out on samples after grinding, polishing and etching in a solution of 2% concentrated nitric acid in ethanol.

An annealing of wiredrawing specimens is carried out at (480-520) °C for different holding times; samples are then quenched in water.

The polished samples are also used for hardness measurements, which were made using a Vickers hardness testing, LEITZ type, at 2Kg load. The measurements are taken from five different indentations. Prior ferrite grain size and complete recrystallization grain size measurements are carried out on five different images using the three circle technique [7].

3. Results and Discussion:

3.1. Microstructure Evolution:

Figure 1a shows the optical microstructure of the material before deformation. It has a homogenous structure of equiaxed ferrite and pearlite with 11 $\mu$m of grain size. However, as a result of deformation (83%) these grains are elongated in the direction of wiredrawing process (Fig.1b). Wiredrawing process followed by recrystallization at different temperatures for different annealing times is needed in order to soft the deformed material. Hence, the optical microstructures of isothermally annealed wires at 480°C for different times reveal the presence of newly small recrystallized grains located at the deformed grain boundaries (Figs.1c and d).
It is observed that grain coarsening started straight away at any portion of the elongated grain boundaries. Prolonged annealing caused growth of the new grains. Both small and large grains are present in the general grain interiors (Fig.1c, d, e and f).

3.2. Primary recrystallization kinetics:

Figure 2 shows a typical curve of the fraction of recrystallized grains ($X_v$) as a function of annealing time at 480°C for the material deformed 83%. It can be seen that the kinetics of primary recrystallization is described by Avrami law. The incubation time is higher than 10000 sec, after 32240 sec 50% of grains are recrystallized. The recrystallization is achieved after 65000 sec. On the other hand, the kinetics of primary recrystallization slows down in later stages.

![Figure 2](image_url)

A typical curve of a plot of $\ln(1/(1-X_v))$ against $\ln t$ for the material deformed 72% is shown in figure 3. This figure indicates that the material gave a linear JMAK plot with a slope of $n = 1.52$. The constants $n$ and $k$ in Avrami
equation as a function of deformation rate are given in table 3. It can be noticed that similar results are obtained for the material deformed 55.5% and 72%. This low value of \( n \) is also found in Nb-micro alloyed steel cold worked 75% [6]. It is suggested that the mechanism of primary recrystallization is slow and is attributed to the presence of second phase [6]. However, the highly deformed material is found to have a higher slope of \( n = 4.05 \). These results could be explained at least by an inhomogeneous distribution of stored energy in less deformed microstructure. As the microstructure of deformed material is not well understood especially with the presence of second–phase, it is difficult to give a general assessment of recrystallization in inhomogeneous microstructure [4].

Figure 4 shows hardness measurements versus annealing time at 480°C for the material deformed 83%. The curve can be divided into two stages. The first stage is recovery, which is the result of the dislocation eliminations; during this short step the material hardness is still high. However, the second stage, which is recrystallization, is quite long and a dramatic softening of the material is observed.

### Table 3. Values of \( n \) and \( k \)

| \( \tau_W \) (%) | \( n \) | \( k \) |
|-----------------|-------|-------|
| 55.5           | 1.56  | 0.033 |
| 72             | 1.52  | 0.019 |
| 83             | 4.05  | 0.014 |

Fig. 4. Hardness versus annealing time: I – recovery, II- recrystallization.
3.3. Activation Energy of primary recrystallization:

The rate of primary recrystallization is given by [4]:

$$\frac{1}{t_{1/2}} = A e^{\frac{Q}{RT}} \quad (1)$$

Where A and R are constants, $t_{1/2}$ is the time for 50% of recrystallized grains at annealing temperature $T(T_{\text{rec}})$ and Q is the activation energy.

Table 4 summarizes values of $t_{1/2}$, $T_{\text{rec}}$ and $\tau_W$ (%). It can be seen that the time, necessary for 50% recrystallization, passes from 304,680 sec to only 1,650 sec when the temperature increases only by 40°C and the deformation rate by 27.5%. This could be explained by the fact that the low deformed microstructure will recrystallize more rapidly than the high deformed microstructure i.e it is attributed to the profound effect of deformation rates.

Table 4. Values of $t_{1/2}$, $T_{\text{rec}}$ and $\tau_W$.

| $T_{\text{rec}}$ (°C) | $t_{1/2}$ (sec) | $\tau_W$ (%) |
|----------------------|----------------|--------------|
| 55.5                 | 504680         | 61900        |
| 500                  | 19700          | 17870        |
| 520                  | 11970          | 2630         |

A plot of $\ln(t_{1/2})$ versus $1/T$ for the material deformed 55.5% is shown in figure 5. It can be seen that there is a good straight line with a slope corresponding to an activation energy of 403.03 KJ mol$^{-1}$.

Fig. 5. $\ln(t_{1/2})$ versus $1/T$.

However, the value of 290 KJ mol$^{-1}$ is found for Fe-3.5%Si deformed 60%[4]. Such activation energies have been found to depend on strain rate and to be lower with the increase in strain rate [4]. The present values of
The activation energy varies from 403.03 KJ mol\(^{-1}\) to 369.82 KJ mol\(^{-1}\) when the strain rate increases from 55.5\% to 83\% (table 5). These values are higher than that for self-diffusion in \(\alpha\)-iron [1].

Table 5. Values of activation energy (Q)

| \(\tau_W\) (%) | Q (KJ mol\(^{-1}\)) |
|---------------|-------------------|
| 55.5          | 403.031           |
| 72            | 391.553           |
| 83            | 369.827           |

The higher activation energy determined for the present work may suggest that the alloying elements in solution inhibit the self-diffusion and control the diffusion drag. However, the present value for the material deformed 83\% is found to be compared to the activation energy for ferrite grain growth of Ti-micro alloyed steel deformed 80\% [8].

### 3.4. Recrystallized grain size:

There is a need to control grain size. The increase in deformation rate contributes in the formation of small grains. A small grain size increases the strength of the material and may also make it tougher for a further wiredrawing. However, a large grain size may be also required in order to increase wiredrawing rates. A relationship between the recrystallized and initial grains size and strain is given by [9]:

\[
d_{\text{rec}} = A \left( \frac{d_0}{\tau_W} \right)^B \tag{2}
\]

Where A and B are constants, \(d_0\) is the initial grain size, \(d_{\text{rec}}\) is the final recrystallized grain size and \(\tau_W\) is the deformation rate.

When \(\ln(t_{1/2})\) is plotted against \(\ln \left( \frac{d_0}{\tau_W} \right)\) a linear relationship is obtained (Fig.6) a good fit to the measured grain sizes, is obtained giving the following equation \(d_{\text{rec}} = 0.16 \tau_W^{-\frac{3}{7}}\). It is necessary to note that as the deformation rate (strain) is increased, the activation energy decreases and consequently the resultant grain sizes are reduced.

![Fig. 6. Variation of \(\ln(t_{1/2})\) versus \(\ln \left( \frac{d_0}{\tau_W} \right)\).](image-url)
4. Conclusion:

The present study deals with the primary recrystallization kinetics of low carbon steel wire. It is appropriate to sum up the main objectives of this study and its achievements.

- This kinetics is found to be described by Avrami law.
- The rate of primary recrystallization is characterized by a gradual increase with increasing the amount of deformation.
- The constants n and k in Avrami law are determined. The value of n is found to increase with highly deformed material while k does not change.
- Annealing process results in softening of the material and producing a uniform and small grain size.
- The activation energy for primary recrystallization is found to be higher for the highly deformed material and is equal to 369.827 KJ.mol\(^{-1}\).
- A relationship between \( d_{\text{rec}} \) and the rate of deformation \( \tau_w \) is established and is the type of:

\[
d_{\text{rec}} = 0.16 \tau_w^{-\frac{4}{3}}
\]

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