Dynamic Recrystallization Behavior of Vanadium Micro-alloyed Forging Medium Carbon Steel

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The dynamical recrystallization of Micro-alloyed forging steel was investigated at deformation temperatures of 850-1150°C and strain rates of 0.01–5 s−1 on a Gleeble-1500 dynamic thermo-mechanical simulator. The stress-strain curves at lower strain rates are typical of the occurrence of DRX and exhibit a peak in the flow stress before reaching steady state. The critical strain for the initiation of DRX has been estimated through the analysis of stress-strain curves and the result showed that the critical strain was correlated to the peak strain by $\varepsilon_c = 0.68\varepsilon_p$. Utilizing the peak stresses $\sigma_p$ measured from the stress-strain curves, constitutive equation governing the dynamic recrystallization has been analyzed and activation energy was determined to be $Q = 379$ kJ/mole, which was significantly larger than that of same composition of V-micro alloyed steel. The grain size was refined from 140 μm to 8–60 μm by DRX. The dynamically recrystallized grain size has been measured and the result showed that logarithm of grain size appeared to be linearly decreasing with the increase in the logarithm of Zener-Holloman parameter $Z = \dot{\varepsilon}\exp(Q/RT)$. However, when the logarithm of grain size was plotted in terms of the inverse of deformation temperature, i.e., $1/T$, the plot showed a significant deviation from the linearity expected from the above linear relationship.

KEY WORDS: dynamic recrystallization; microstructure evolution; forging; medium carbon steels.

1. Introduction

Dynamic recrystallization (DRX) is the most important restoration mechanism during hot deformation of austenite; affecting the final microstructure and, therefore, the mechanical properties of the deformed material. Extensive research has been performed on the mechanisms and the variables involved in this phenomenon.1–5) These mechanisms are believed to vary depending on the steel composition and deformation conditions. However, in commercial low-carbon steels, transformation usually restricts the study of DRX of austenite at lower temperatures and, hence, limits the study of this phenomenon over a wide temperature range. Austenitic stainless steels, which do not undergo phase transformation over a wide temperature range, can be used as model alloys to investigate recrystallization.

During deformation, the pre-existing grain boundaries elongate along the deformation direction, grain boundary serration appears, and then new DRX grains nucleate at the serrated pre-existing grain boundaries. The general descriptive model for DRX is that nucleation occurs at the serrated pre-existing grain boundaries and increases until a layer of DRX grains covers these boundaries. Then the recrystallization reaction proceeds via nucleation at the interfaces between the recrystallized and non recrystallized material, until new grains consume the structure. This type of DRX structure has been known as a necklace structure. Once the necklace structure is completed, a steady state is reached; continuing nucleation and growth maintain the structure (with equiaxed grains) at a constant stress.

Despite this simple explanation for DRX phenomenon (i.e., necklacing), its evolution during hot deformation of different materials is not simple and different parameters, such as strain rate, temperature, and initial grain size can affect the necklace structure.6–10) Although this structure is observed during hot deformation of many materials and under different deformation conditions,11–13) there are still many issues regarding the progress of the DRX microstructure based on this phenomenon and especially the contribution from other DRX mechanisms, such as grain boundary sliding and continuous DRX. Since the mechanical properties of most metallic materials strongly depend on their microstructures, the studies on mechanisms for microstructure evolution during thermomechanical processing are of great practical importance. An important mechanism for the microstructure control is dynamic recrystallization (DRX),14–16) which frequently takes place under hot working of materials with low to medium stacking fault energy. The general characteristics of DRX, such as the effect of deformation conditions on the dynamic grain structures evolved and the flow stresses, etc., have been fairly clarified for various conventional metals and alloys.

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In this present research, the effects of thermomechanical parameters including strain, strain rate, and temperature on both mechanical and microstructural aspects have been investigated. The main focus has been on the effect of vanadium micro-alloy on DRX behavior. The dynamic recrystallization and recrystallization grain size behavior of vanadium micro-alloyed forging steel at different temperature at constant strain rate has been investigated. Temperature dependency of recrystallized grain size was investigated thoroughly.

2. Constitutive Equations

The flow stress ($\sigma$) during hot working in steels and alloys depends on two deformation control variables, temperature ($T$) and strain rate ($\dot{\varepsilon}$), through the Zener–Hollomon parameter ($Z$), which is a generally accepted method since 1944 to characterize the constitutive relationship during thermal deformation.\(^{17}\)

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) \quad \text{.............................. (1)}$$

$$\sigma = \sigma(Z, \dot{\varepsilon}) \quad \text{........................ (2)}$$

Where $Q$ is the activation energy for hot deformation, and gives information of materials; $R$ is the gas constant ($8.314$ Jmol$^{-1}$ K$^{-1}$); $\dot{\varepsilon}$ is strain rate and $T$ is the absolute temperature at which deformation occurs.

Generally, there are three widely accepted relations between flow stress and $Z$:

1) The favorite equation of creep (low stress) is the power law:

$$Z = A_1 \sigma^n \quad \text{............................. (3)}$$

where $\sigma < 0.8$ which is also suitable for the case at high temperature and low strain rate;

2) Formerly hot working analysts (high stress) favored the exponential law:

$$Z = A_2 \exp(\beta \sigma) \quad \text{........................ (4)}$$

where $\beta > 1.2$ which is also applicable for the case at low temperature and high strain rate;\(^{19}\)

3) A function, which includes both two equations above in the limits, was introduced in the 1960s by Sellars and Tegart:\(^{19}\)

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) = A\left[\sinh(\alpha \sigma)\right]^n \quad \text{for all $\sigma$} \quad \text{...... (5)}$$

Where coefficients $\alpha, \beta, n$ ($n_1$), and $A(A_1, A_2)$ are material constant. The constants $\alpha$ and $n_1$ are related by $[\beta = \alpha n_1]$ so that $\alpha$ and $n_1$ can be simply determined from experimental data at high and low stresses. The hyperbolic law of Eq. (5) gives better approximations between $Z$ and stress than Eqs. (3) and (4), whatever the value of the stress and the type of hot working. Therefore Eq. (5) is widely used to establish the constitutive relationship between stress and control variables $T$, $\dot{\varepsilon}$ for a certain metal.

3. Materials and Methods

The material used in this investigation was the commercial Vanadium micro-alloyed medium carbon steel, and its chemical composition in (wt.%) is given in the Table 1. An ingot of medium carbon Vanadium Micro alloyed steel of thickness 250 mm was cast in a vacuum induction furnace. This ingot was homogenized for 1 hour at 1250°C and was subjected to hot size rolling at 1250°C to produce a slab of thickness 70 mm. This slab was further subjected to usual hot rolling procedures to produce a final plate of thickness 13 mm. Rod-shaped specimens of 10 mm in diameter and 15 mm in length were machined from the plate for hot-deformation experiments. The rod-shaped specimens were taken with their length parallel to the rolling direction.

The single hot compression tests were carried out on the Gleeble 1500 thermo-mechanical simulator. A Gleeble is a dynamic testing machine that can simulate a wide variety of thermal/mechanical/metallurgical situations. Starting with the basic treatment of metals to obtain specific structures and proceeding through the testing of specimens taken from finished products, the Gleeble can simulate and provide test data on almost any thermal/mechanical exposure the material sees during its life. Therefore, hot working in steels and alloys depends on two deformation control variables, temperature ($T$) and strain rate ($\dot{\varepsilon}$), through the Zener–Hollomon parameter ($Z$), which is a generally accepted method since 1944 to characterize the constitutive relationship during thermal deformation.\(^{17}\)

4. Results and Discussions

4.1. Hot Deformation Flow Curves

The stress strain curves for deformation at the temperatures of interest (1150, 1050, 950 and 850°C) are shown in Fig. 1. As expected, for a given strain rate, the flow stress increases with a decrease in temperature. All curves show initial work hardening but only a few deformation conditions develop a clear peak indicative of dynamic recrystallization (DRX) in the flow curve. These peaks occurred under high temperature, low strain rate combinations (low $Z$ values). In general the flow stress increases with strain rate, but as the temperature decreases the changes in strain rate have less influence on the flow stress and the shapes of the flow curves become almost identical. The strain rates that appear to be having the most influence on the flow

<p>| Table 1. Chemical composition in weight percentage of vanadium micro-alloyed steel. |
|---------------------------------|---|---|---|---|---|---|---|---|---|---|---|</p>
<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cu</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Al</th>
<th>N</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>S45CVMn</td>
<td>0.45</td>
<td>0.15</td>
<td>1.20</td>
<td>0.03</td>
<td>0.04</td>
<td>0.3</td>
<td>0.2</td>
<td>0.1</td>
<td>0.05</td>
<td>0.08</td>
<td>0.02</td>
<td>0.006</td>
<td></td>
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stresses (0.01 and 0.1 s\(^{-1}\)) are those that are typical of a laboratory environment, while the more industrially relevant rates show little influence on the material flow stress.

At low strain rates (i.e., \(\varepsilon \leq 0.01\) s\(^{-1}\)), the typical sequence of a peak stress followed by a steady state in the flow curves implied that DRX has been the dominant restoration process; although, again it was not a true steady state as some softening continued. As the strain rate increased above 1 s\(^{-1}\), the flow curves showed a broader peak followed by no, or limited, steady state. The deformation heating generated in these conditions (i.e., high strain rates) is the most likely cause for the shape of these flow curves.

4.2. Work Hardening Curves

The DRX starts at a critical strain that can be determined either by flow curve analysis or through microstructure observations. Flow curve analysis is a simpler and quicker method compared to the microstructural analysis, as the latter needs a large quantity of samples to be examined and it can be difficult to precisely ascertain the new grains. The flow curve analysis has been used in the current work to define the critical strain \(\varepsilon_c\) for the start of DRX. In some cases, though, the results were confirmed by microstructure observations. The basis of the flow curve analysis method is that DRX (as a restoration process) affects the flow curve shape by changing the rate of work hardening through the introduction of new strain free grains. A mathematical technique firstly proposed by Kocks and Mecking\(^{20}\) and then continued by McQueen and Ryan\(^{21,22}\) can be used to highlight this point in the flow curves. In this method, the onset of DRX corresponds to a deviation in the work hardening curves, i.e., the differentiation of stress with respect to the strain \(\theta = \frac{\partial \sigma}{\partial \varepsilon}\).\(^{23}\) It should be noted that the critical stress and strain in this method corresponds to a point where a certain fraction (at least 2 pct) of DRX has occurred in the microstructure to make a noticeable change in the flow curve.

The work hardening rate \(\theta = \frac{\partial \sigma}{\partial \varepsilon}\) was calculated from the flow curve and plotted as a function of stress (Fig. 2). These work hardening curves consisted of two stages. In the first stage, the work hardening rate decreased rapidly with increasing stress, possibly due to the dynamic recovery, until the start of the second stage. At this point, a change occurred in the slope of the curve and DRX was considered to have started. The extrapolation of the second stage in the work hardening curve (where the fluctuations related to DRX have started), to \(\theta = 0\) establishes the saturation stress \(\sigma_s\), which would correspond to the softening due to recovery alone.\(^{21}\) The difference between this saturation stress and the flow stress obtained from the flow curves will determine the softening due to DRX. The slope change in the work hardening curves is used to identify the critical stress \(\sigma_c\) and strain \(\varepsilon_c\) for initiation of DRX. It is apparent in Fig. 2 that the critical stress derived from the work hardening curves (the slope change point) lies on a specific line at different deformation conditions.

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Fig. 1. True Stress-True Strain curves at various temperatures and strain rates (a) 0.01 s\(^{-1}\) (b) 0.1 s\(^{-1}\) (c) 1.0 s\(^{-1}\) and (d) 5.0 s\(^{-1}\).

Fig. 2. Work hardening curves of samples deformed at different deformation conditions.
It is difficult in above mentioned method to obtain the exact value for the inflection point in the work hardening curves, and an analytical method was also used to detect this point. In this approach, used by Poliak and Jonas, the second numerical derivative of flow curves as a function of stress (i.e., $\frac{\partial^2}{\partial \sigma^2} \ln \sigma$ vs $\sigma$) has a minimum at the inflection point of work hardening curve. This unique minimum point gives a good estimate of the critical stress (Fig. 3). The corresponding strain $\varepsilon$ can be extracted from the original flow curve. It is clear that this approach gives a much more easily defined value for $\sigma_c$, but does not give an estimate of $\sigma_c'$. Therefore, both methods are required for determination of the two important stress values.

4.3. Apparent Activation Energy

As shown in the stress–strain curves, increasing strain rate and decreasing deformation temperature can make the flow stress increase and hamper the occurrence of DRX. The relationship can be described by Zener–Hollomon parameter.

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) \quad \ldots (10)$$

Here, $Q$ is activation energy (kJ/mol) for hot deformation, $R$ the universal gas constant and $T$ is the absolute temperature.

According to Eq. (10), the value of $Z$ is bigger at higher strain rate and lower deformation temperature. Its change law is accordance with the variety of peak stress. The relationship of peak stress and $Z$ is usually expressed by one generally accepted hyperbolic sine function as following.

$$Z = A \left[\sinh \left(\alpha \sigma_p\right)\right]^n \quad \ldots (11)$$

Here, $A$ and $\alpha$ are the material dependent constants, $n$ the stress exponent and $\sigma_p$ the peak stress.

According to Eqs. (10) and (11), we have

$$\dot{\varepsilon} = A \left[\sinh \left(\alpha \sigma_p\right)\right]^{n} \exp\left(-\frac{Q}{RT}\right)$$

Taking natural logarithms then partial derivative on both sides of Eq. (12), we have

$$\frac{\partial}{\partial (\ln \dot{\varepsilon})} \ln \dot{\varepsilon} = \frac{n \partial}{\partial (\ln \sigma_p)} \left[\ln \left(\sinh \left(\alpha \sigma_p\right)\right)\right] - \frac{Q}{RT} \frac{\partial}{\partial (1/T)}$$

If $T$ is constant, Eq. (13) can be rewritten as

$$\frac{1}{n} = \frac{n}{\partial (\ln \sigma_p)} \left[\ln \dot{\varepsilon}\right]$$

The high stress and low stress equations (Eqs. (3) and (4)) that the slope of the plot $\ln \varepsilon$ against $\ln \sigma_p$ and the slope of the plot of $\ln \varepsilon$ versus $\sigma_p$ can be used for obtaining the value of $n'$ and $\beta$, respectively. These plots are shown in Figs. 4(a) and 4(b). The average value of $n'$ and $\beta$ was calculated by

Fig. 3. Factor of $-\frac{\partial^2}{\partial \sigma^2}$ as a function of flow stress at different deformation conditions.

Fig. 4. Relationship between: (a) $\ln \sigma$ and $\ln (\dot{\varepsilon})$; (b) $\sigma$ and $\ln (\dot{\varepsilon})$; (c) $\ln \sinh(\alpha \sigma_p)$ and $\ln (\dot{\varepsilon})$; (d) $\ln \sinh (\alpha \sigma_p)$ and $\ln (\dot{\varepsilon})$. 
the linear regression method and the value was found out to be 8.961376 and 0.091494, respectively. This gives the value of $\alpha = \frac{\beta}{\beta_1} = 0.010209$. Which is consistence with the comparable results ($\alpha = 0.015$) for similar composition of vanadium micro alloyed steels$^{16}$ and ($\alpha = 0.012$) for non-vanadium micro-alloyed steels.$^{25}$

So, there is a linear relationship between $\ln \sinh(\alpha \sigma_p)$ and $\ln \varepsilon$ when $T$ remains constant. According to the relationship curves of $\ln \sinh(\alpha \sigma_p)$ and $\ln \varepsilon$ shown in Fig. 4(c), the value of $n$ can be estimated because $n$ is equal to the reciprocal of linear slope. It shows that the value of $n$ increases with the increase of deformation temperature. And the average value of $n$ has been calculated 4.2–5.1 for different non-vanadium micro-alloyed steels.$^{25}$

And according to Eq. (13), there is a linear relationship between $\ln \sinh(\alpha \sigma_p)$ and $\ln \varepsilon$ when $T$ is constant,

$$Q = R \times n \times \left[ \frac{\partial}{\partial T} \ln \left( \frac{\alpha \sigma_p}{\sqrt{T}} \right) \right]$$

According to the relationship curves of $\ln \sinh(\alpha \sigma_p)$ and $\ln \varepsilon$ shown in Fig. 4(d), the linear slope increases with the increase of strain rate, the average value of the slopes is 6.745635. Because the value of $n$ has been calculated, the average value of $Q$ can be obtained according to Eq. (15) and it is 379 kJ/mol. The results (Fig. 4(d)) showed that the activation energy, which is significantly larger than that (270–320 kJ/mol) reported in V-microalloyed medium-carbon steels.$^{26-28}$ The distinctively large activation energy suggests that dynamic recrystallization more strongly interacted with particles in the present alloy system, because AlN particles, in addition to the VN particles, could precipitate in the present alloy. This result was published in our previous paper.$^{29}$ The apparent activation energies associated with this equation for non vanadium micro alloyed steels are range from 312 to 326 kJ/mole for the base, Nb, and Nb–B steels. The higher value of 382 kJ/mole determined for the Cu–Nb–B steel is probably due to the high level of copper in solution.$^{25}$ The hot working activation energy depends on the material being considered, it is usually referred to as apparent value, because no account is generally taken of the internal microstructural state and it is only derived from an Arrhenius plot with a linear range and the assumption that the microstructure remains constant.

### 4.4. Critical and Peak Strain and Peak Stress

At a constant initial grain size, the constitutive equations of peak and critical strains are usually shown as power-law functions of $Z$ in the form of$^{20}$

$$\varepsilon = AZ^n \quad \ldots \quad (6)$$

where $A$ and $n$ are constants.

The peak and critical strains derived from the flow curves were plotted on a logarithmic scale with respect to $Z$ (Fig. 5). With estimation of the values of $A$ and $n$, similar type equations were developed for both peak ($\varepsilon_p$) and critical ($\varepsilon_c$) strains:

$$\varepsilon_p = 8.146 \times 10^{-3} \times Z^{0.107} \quad \ldots \quad (7)$$

$$\varepsilon_c = 5.456 \times 10^{-3} \times Z^{0.112} \quad \ldots \quad (8)$$

These equations indicate that the critical strain is a fraction of the peak strain ($\varepsilon_c = 0.68 \varepsilon_p$). A nearly similar constant ratio between critical and peak strains has been reported for stainless steel,$^{30}$ copper,$^{31}$ C–Mn steels, and Fe–Ni–C alloys.$^{32}$

The peak flow stress ($\sigma_p$) also showed a power-law function with Zener–Hollomon parameter in the form of (Fig. 6)

$$\sigma_p = 2.462 \times Z^{0.15} \quad \ldots \quad (9)$$

### 4.5. DRX Gain Size and Microstructure Evolutions

The dynamic recrystallization of grain size ($d_{\text{DRX}}$) depends on the deformation temperature and strain rate, which are the functions of the $Z$ parameter alone and independent of the initial grain size.$^6$ By fitting a linear regression to the experimental results in Fig. 7, the equation for the depen-

**Fig. 5.** Critical and peak strains as a function of Zener-Holloman parameter.

**Fig. 6.** Peak stress as a function of Zener-Holloman parameter.

**Fig. 7.** DRX grain size as a function of Zener-Holloman parameter.
dence of the dynamic recrystallization grain size ($d_{drx}$) on the $Z$ parameter is determined to be:

$$d_{drx} = 1.204 \times 10^2 \times Z^{-0.056} \quad (\text{16})$$

Which indicates that $\ln(d_{drx})$ linearly decreases with increasing $\ln(Z)$. Thus the optimal process parameters to obtain finer grain size during hot deformation can be determined by Eq. (16).

**Figure 8** compares optical micrographs of austenite grain structure between the two prior-cooling-conditions. The grain size ranged from about 8 $\mu$m at low temperature to about 60 $\mu$m at high temperature depending on the strain rate. Considering the initial grain size (about 140 $\mu$m), dynamic recrystallization brought about a great deal of grain refinement, particularly at low temperature and fast strain rate. There is strong evidence to link the recrystallized microstructure to the deformation conditions of temperature and strain rate ($Z$ value). The elongated necklace structure and its evolution during deformation vary with the deformation conditions, and this type of structure is not clearly visible for all deformation conditions. The clearest elongated necklace structure and subsequent evolution of the DRX microstructure occurred at moderate $Z$ values (e.g., deformation at 800°C and 0.1 s$^{-1}$ and 5.0 s$^{-1}$ in **Fig. 9(a) and 9(c)**). With an increase in the temperature at constant strain
rate, the bulges started to separate from the original boundaries and formed new grains. At the 950°C and 0.1 s\(^{-1}\) and 5.0 s\(^{-1}\) (Figs. 9(b) and 9(d)), most of these original boundaries were decorated by DRX grains. At this stage, the first layer of the necklace structure has formed. It has been shown\(^{(13)}\) that the orientation of these new grains, which formed through bulging of original grain boundaries, is very similar to the orientation of the initial grains. This necklace structure was completed at a strain far beyond the start of the steady-state strain and replaced all of the original deformed microstructure.

However, when the logarithm of grain size was plotted in terms of the inverse of deformation temperature, \(i.e. 1/T\), the plot showed a significant deviation from the linearity expected from the above linear relationship (Fig. 10). At low temperature, the microstructures showed were elongated grain boundaries, but in case of high temperature microstructures were equiaxed grain boundaries. The dynamically recrystallized grains started to form at lower (800°C) temperature. As temperature start increasing after 950°C, DRX grains showed another value. When the optical micrographs are critically analyzed, it was found that below 950°C, DRX grains are elongated, whereas above 950°C the grains are equi-axed in nature.

5. Conclusions

The hot deformation behavior of Vanadium micro alloyed steel was investigated to characterize the evolution of the DRX structure. The effect of different deformation parameters such as strain, strain rate, and temperature were investigated. The key finding of the investigation, however, can be summarized as follows:

1. The logarithmic of DRX grain size was showed a significant deviation from the linearity expected \(i.e. (d_{ave} = 1.204 \times 10^2 \times Z^{0.050})\) when plotted in terms of the inverse of deformation temperature, \(i.e. 1/T\). This deviation was due to at low temperature below 950°C, the DRX grains showed elongated necklace structure. As the temperature start increasing, the grains showed an equiaxed structure.

2. Using the hyperbolic sine law, the activation energy of hot deformation of studied steel is calculated and is equal to 379 kJ/mol.

3. The method developed by Poliak and Jonas provided precise results to determinate the critical conditions for the initiation of DRX from the inflection point in the strain hardening rates versus stress relationship of present vanadium microalloyed steels.

4. The peak and critical strains (derived from flow curve analysis) showed power-law functions with the Zener - Hollomon (Z) parameter and the ratio of critical strain to peak strain was \(\sim 0.68\).

5. The average value of \(n\) or slope of lines \(\ln\varepsilon\) versus \(\ln\sinh(\sigma_{cr})\) for the studied steel in the temperature range 850–1150°C and strain rate range 0.01–5 s\(^{-1}\) is 6.758697.

**REFERENCES**