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A JMAK-microhardness model for quantifying the kinetics of restoration mechanisms in inhomogeneous microstructure

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Abstract

The microstructure of heavily drawn wire is inhomogeneous and subsequent annealing of the material results in inhomogeneous recrystallization. Standard JMAK analysis of the kinetics of recovery and recrystallization for such microstructure is cumbersome and sometimes unreliable. This investigation was carried out on wire drawn (to true strain of 2.31) oxygen free high conducting (OFHC) copper, which was subsequently annealed at 250, 400 and 500 °C for annealing times ranging from 10 s to 1 h. The microstructural changes during the annealing were characterized by optical and scanning electron microscopy, orientation imaging microscopy (OIM) and microhardness. While standard JMAK analysis can be used to analyze the kinetics of recrystallization and grain growth, it was inadequate for quantifying the recovery kinetics. However, the modified JMAK-microhardness model developed enabled us to evaluate the kinetics of recovery, recrystallization and grain growth, using a single equation. In this approach, the JMAK model is expressed in terms of microhardness data, from which the parameters of the different restoration kinetics were determined. The values of JMAK exponent, n, the temperature-dependent constant, k, and the activation energy, Q, for recovery, recrystallization and grain growth obtained by the new method compared well with values in the literature.

Keywords: Recovery; Recrystallization; Grain growth; Kinetics; Wire drawn; OFHC copper

1. Introduction

The processing of cast metals and alloys usually involves a sequence of deformation and annealing procedures. Alteration of the sequence or procedure employed in processing can be used to control the microstructure, which in turn affects the property of the material. The microstructural changes which occur during such processing depend on the degree of the restoration mechanism; i.e. recovery, recrystallization and grain growth. Although recovery and recrystallization usually precede grain growth, the three processes generally overlap, and it is often difficult to unambiguously distinguish between these phenomena [1]. Traditionally, the kinetics of each of the three restoration mechanisms is usually treated and analyzed separately with different equations. A brief review of the kinetics of the three mechanisms is presented below.

1.1. Recovery kinetics

During recovery, some of the stored strain energy is relieved by any of the recovery mechanisms, which include dislocation annihilation, dislocation rearrangement and subgrain growth. Because microstructural changes that occur during recovery are subtle, recovery is generally measured indirectly by the changes in hardness, yield stress or resistivity. In spite of the difficulty in discerning microstructural changes during recovery, some empirical relationships based on microstructure have been developed for the kinetics of recovery [2,3]. The most common relationship is expressed in terms of the dislocation density, ρ, and is given as [2,3]:

$$\frac{\mathrm{d}\rho}{\mathrm{d}t} = -\kappa\rho^{p} \tag{1.1}$$

where κ is the velocity at time t or reaction rate and p is the order of reaction and has been determine to be 2 for LiF and 3 for copper and nickel [2,3]. Therefore, analysis of recovery kinetics requires a quantitative measurement of the dislocation density

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by transmission electron microscopy (TEM), a task which is extremely laborious.

1.2. Recrystallization kinetics

The generally accepted empirical rate equation that most describe the isothermal kinetics of recrystallization requires measurement of the recrystallized volume fraction, X_v, and can be can be expressed as [4]:

$$\frac{dX_v}{dt} = n\kappa^n t^{n-1}(1 - X_v) \tag{1.2}$$

where n is the order of the reaction, and the factor $(1 - X_v)$ may be regarded as an allowance for retardation in reaction rate due to impingement. Assuming κ and n to be true constants independent of X_v (and thus of t) for a given temperature, the integration of Eq. (1.2) yields:

$$\ln \frac{1}{1 - X_v} = (\kappa t)^n \tag{1.3}$$

which yields a sigmoidal rate curve, and can be also expressed as [5–7]:

$$X_v = 1 - \exp[-kt^n] \tag{1.4}$$

Eq. (1.4) is attributed to Kolmogorov [5], Johnson and Mehl [6] and Avrami [7], and is commonly known as JMAK. The JMAK equation is strictly valid only when the recrystallized grains are distributed randomly and when the grains are growing independently of one another [8]. The order of the reaction n, also known as the Avrami or JMAK exponent, reflects the nucleation rate and/or the growth morphology, and k is the pre-exponential factor, a kinetic parameter depending on the annealing temperature, nucleation rate and growth rate. In general, n takes the value of $1 \leq n \leq 2$ for one-dimensional growth, $2 \leq n \leq 3$ for two-dimensional growth and $3 \leq n \leq 4$ for three-dimensional growth [4]. For no change in mechanism, n is insensitive to temperature, whereas k is the temperature-dependent constant expressed as [7]:

$$k = k_0 \exp\left(-\frac{Q}{RT}\right) \tag{1.5}$$

where k_0 is a constant, Q the activation energy, R the gas constant ($R = 8.314472\ \mathrm{J\ mol^{-1}\ K^{-1}}$) and T is the absolute annealing temperature.

Even if one vaguely separates recovery from recrystallization, it is still problematic to precisely determine the volume fraction of recrystallized material, X_v, by the conventional methods in inhomogeneous recrystallized microstructure. The most widely used conventional method is that of quantitative metallography performed under optical microscopy on a series of samples recrystallized to different extents [9–11]. Etching technique is generally used to reveal the grain structure and a number of micrographs are taken of randomly selected areas. A point-counting technique is then applied to obtain average values of the recrystallized fraction [9,12]. However, this method is rather time consuming, depends on the image contrast of the micrographs, and may not yield accurate data from inhomogeneously deformed microstructure such as that due to drawing. Recent studies [13–15] on drawn and annealed copper wires have shown that the inhomogeneity of the microstructure across the wire, due to inhomogeneous deformation during drawing, is visible as three distinct concentric regions; the inner core, the mid-section, and the outer surface. While the mid-section is noted to exhibit the highest recrystallization rate, the inner core has the lowest. This results in inhomogeneous recrystallization across the wire. At low annealing temperature, recrystallization was observed to initiate at the mid-section [14,15], and at high annealing temperature, abnormal grain growth emerged at the mid-section [15,16]. Such inhomogeneities pose difficulties in quantifying the recrystallized volume fraction by the conventional metallographic technique. Furthermore, this method is limited by the resolution of the optical microscopy in which the smallest recrystallized grain size that can be clearly observed is of the order of 2–5 μm. Needless to say metamorphosis, in which apparently unrecrystallized regions begin to appear recrystallized and vice versa, creates additional problems when analyzing the microstructure under polarized light [17].

1.3. Grain growth kinetics

The kinetics of grain growth, on the other hand, requires a quantitative measurement of the grain size with time. According to Burke and Tunbull [18], the rate equation for a growing grain is expressed as

$$\frac{dR}{dt} = kR^{-(n-1)} \tag{1.6}$$

where R is the mean grain radius at time t. Empirically, exponent n is between 1.5 and 4 [19], and theoretical grain growth models give $n = 2$ [8,20]. Eq. (1.3) is of a similar form to the kinetics of sub-grain growth [8].

The problems associated with quantifying the recrystallization microstructure resulting from severely deformed materials have motivated the present work. The current method for analysis requires that the recovery and recrystallization kinetics are evaluated separately, and this often involve the use of different quantification techniques and equations for each of these restoration processes. Here, a modified JMAK model for evaluating the kinetics of recovery, recrystallization and grain growth in drawn and annealed OFHC Cu is proposed. The model incorporates microhardness data, instead of relying on microstructure, and provides a unified equation, that can be used to accurately determine the parameters for the kinetics of recovery, recrystallization and grain growth.

2. Experimental procedure

The OFHC Cu (99.99%) wires used in this study were drawn at room temperature, at a controlled rate of 10 mm/s, to a true strains, ε of 2.31 [where $\varepsilon = 2\ln(d_0/d)$, d_0 and d are the initial and final diameters, respectively]. Specimens of the drawn wires were isothermally annealed at 250 °C (specimen A), 400 °C (specimen B) and 500 °C (specimen C) for dura-