TEMPERATURE INDEPENDENT TIME EXPONENT FACTOR IN COPPER RECRYSTALLIZATION PROCESS

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Abstract

Isochronal annealing was followed by the two adopted methods, microhardness, and metallographic observation for studying the kinetics of recrystallization in pure copper. From these it was found that nucleation of pure copper started at 300°C. The changes in microhardness during the isothermal annealing were measured at temperatures 250, 300 and 350°C. For these temperatures, the complete recrystallization as being determined from the metallographic observation, was found to be achieved after times of 5, 1 and 0.5 hours. The kinetics of the recrystallization process of this material was studied using an equation of the form.

$$x = 1 - \exp(-Kt^n)$$

where (x) is the fraction of the process completed at a given time t, k is the rate constant, and (n) is a constant, characteristic for such processes. The time exponent (n) was found to be the same for all chosen temperatures, i.e., it seems to be temperature independent constant. On the other hand, the recrystallization rate constant (K) was found to be a temperature dependent one, where it increased as the temperature increased, and having the values of 4 x 10^{-5}, $2x10^{-4}$ and 7.5 x 10^{-4} for the temperatures 250, 300 and 350°C, respectively. The corresponding activation energy for the recrystallization process was calculated, using the Avrami equation and it was found to be (1.47 ev).

Introduction

Several authors have studied in detail the kinetics of recovery and recrystallization of single crystals of polycrystalline copper and dilute copper alloys [1-6]. They showed that there are several factors which can affect the rate of the recrystallization process, .e.g. the concentration, the type of deformation (pre-straining, cold or hot rolling, etc.), as well as temperature and time.

According to the overall softening arises from the linear combination of static recovery, classical recrystallization and metadynamic recrystallization. The recrystallized components are described by a general law of the form :

$$X = 1 - xp \ (-Kt^n) \ \ldots\ldots\ldots\ldots \tag{1}$$

where x is the volume fraction of the material that has completed the process, K is the rate constant and n is the time exponent. This expression is simply the general form of the Johnson-Mehl equation, which was originally proposed for the formation of pearlite from austenite [7], but also adequately describes recrystallization [8-10]. The purpose of this work is to analyze the parameters in Eq. (1). The activation energy was calculated according to Avrami's equation [11,12].

$$\text{Rate} = Ae^{-B/T} \ \ldots\ldots\ldots\ldots\ldots \tag{2}$$

where A and B are constant using

$$\text{Rate} = 1/ \ (\text{time for 50 \% recrystallization})\ldots\ldots \tag{3}$$

2. Experimental Procedure :

The chemical composition of the samples is shown in Table 1. Specimens of dimensions I cm x I cm were cut form sheets of thickness 0.1 cm for both isochronal and isothermal studies. The specimens were annealed at temperatures ranging from 20–600°C, for 2 hours in a special designed automatically controlled oven, the temperature was measured to an accuracy ± 2°C using a thermocouple mounted near the sample.

Microhardness measurements were carried out using Shimadzu microhardness tester of type M (Kyoto-Japan). The applied load was

50 g, for 5 sec, and more than 10 readings were tried at each temperature and their mean values and standard deviation were calculated for each condition.

RESULTS

The isochronal annealing curve is as shown in Fig. 1, while the isothermal annealing curves at 250, 300 and 350°C are as shown in Fig. 2. The degree of recrystallization (x) can be calculated by using the equation.

$$x = x_t / x_R ,$$

where x_t is the amount of recrystallization % at (t), and x_R is the complete recrystallization which was considered as unity. The value of the exponent (n) in Eq. (1) can be determined by plotting log ln [x_R / ($x_R - x_t$)] against log t as shown in Fig. 3 (for simplicity x_R / ($x_R - x_t$) is represented as θ). The value of the rate constant was obtained from plots of ln θ versus t^n as shown in Fig. 4.

To determine the activation energy of the recrystallization process, the recrystallization percentage versus the time of annealing was plotted for temperatures studied as shown in Fig. 5, then the relation between log (rate of recrystallization) versus 1/T was plotted as shown in Fig. 6, where the rate of recrystallization was taken as 1/(time for 50% recrystallization). The microstructure of partially recrystallized and complete recrystallized pure Cu is shown in Fig. 7 and Fig. 8, respectively

Discussion :

Inspection of the results in Fig. 1 in view of the metallographic investigations gives us the impetus that, nucleation of the studied material starts at 300°C, while it was previously reported that complete recrystallization occurred after annealing at 250° – 350°C depending on the impurity concentration [13-16].

In order to define the factors affecting the recrystallization process, analysis of the experimental data according to the general rate
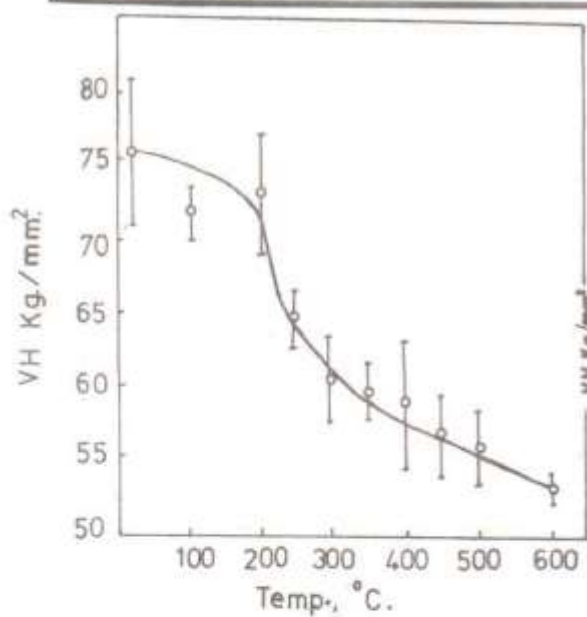
Fig. 1:   Isochronal annealing of pure copper.
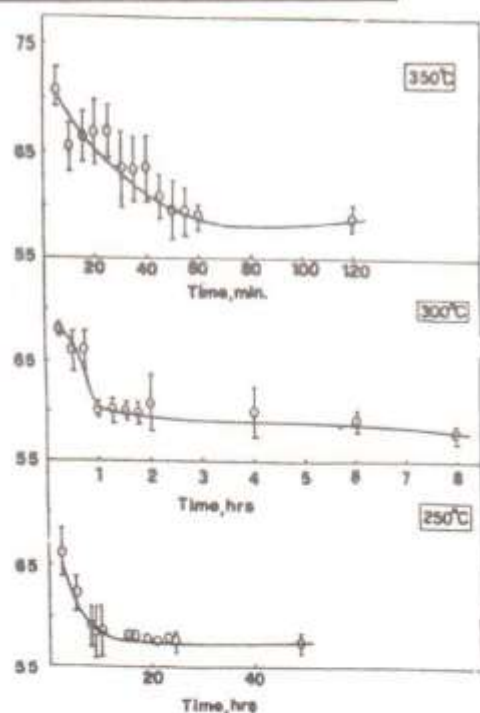

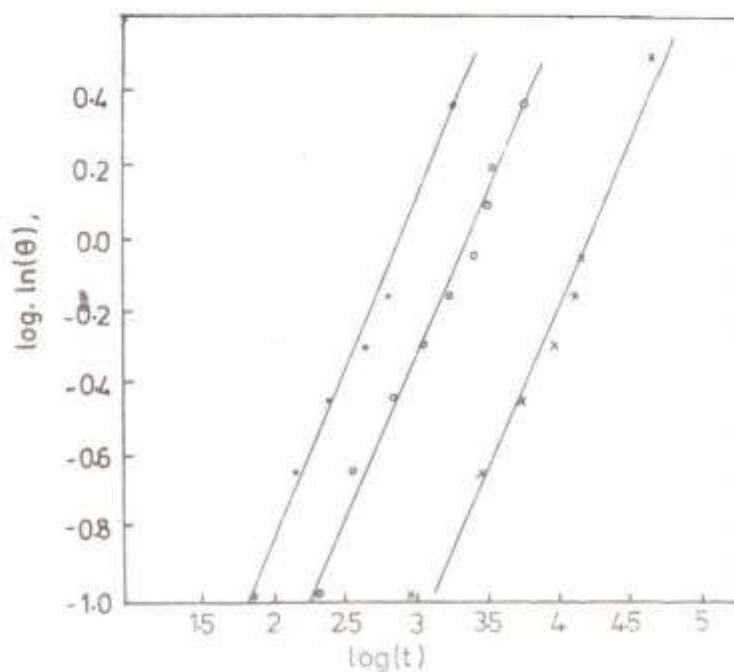Fig. 2:   Isothermal annealing of pure copper.

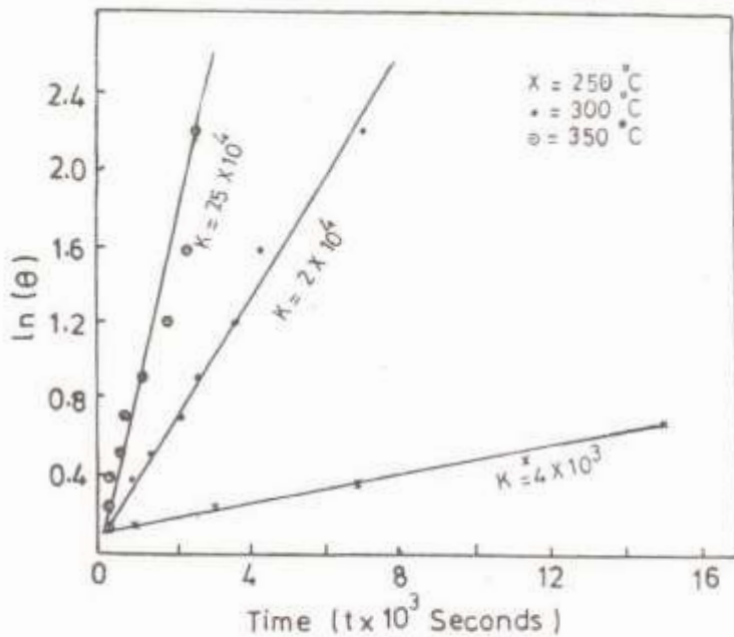Fig. 3:   Graphical determination of the time exponent (n).

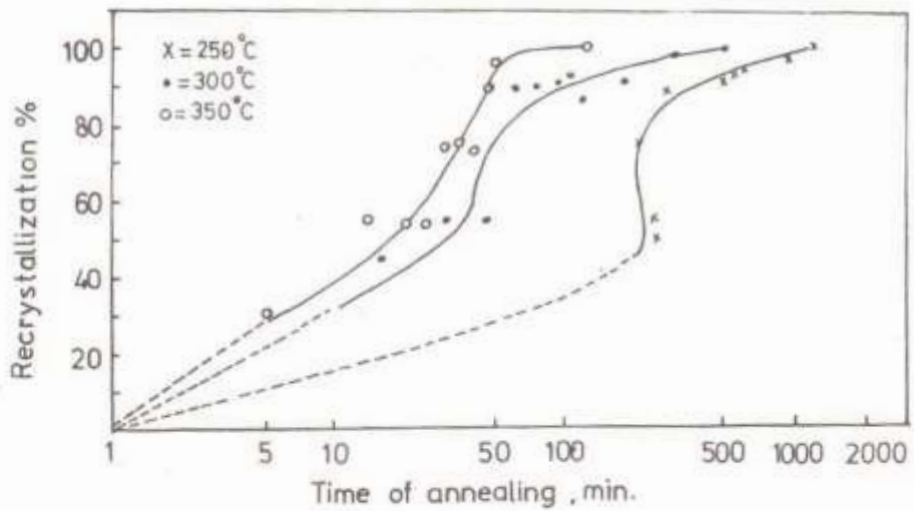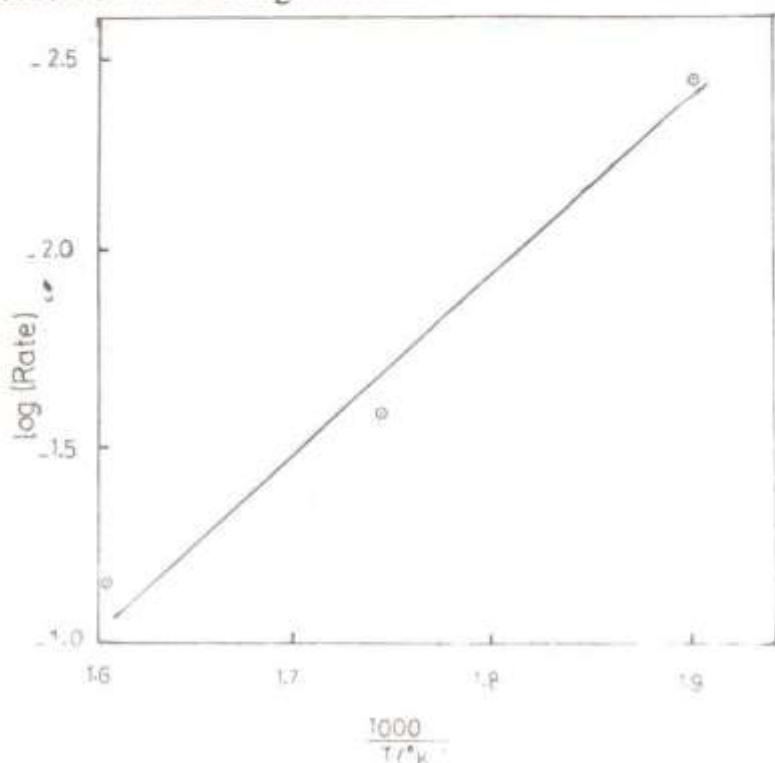Fig. 4: Graphical determination of the rate constant K.

Fig. 5: Isothermal annealing of pure copper at temperatures 250, 300 and 350°C.

law of the form of Eq. (1) was done. Such analysis shows that :

i) The time exponent (n) is equal to 1 at all the tested temperatures (Fig. 3). This means that it is temperature independent, in good agreement with that obtained for metadynamic recrystallization of polycrystalline copper [3].

ii) The lower value of the time exponent in the present study in comparison with those reported earlier, may be attributed to the fact that it decreases as long as all nucleation sites are saturated at the initial stages of the process [17].

iii) The rate constant (K) value is found to increase rapidly with increasing temperature, i.e., it is temperature dependent (Fig. 4).

iv) Analysis of Eq. (2) was followed by metallographic observation, the calculated activation energy is found to be (1.47 e.v.) for the recrystallization process and that reported earlier for copper varied between (0.72–1.1 e.v.). The noticeable difference in the activation energy value seems to be due to the differences in the condition of the starting material and the other factors affecting it
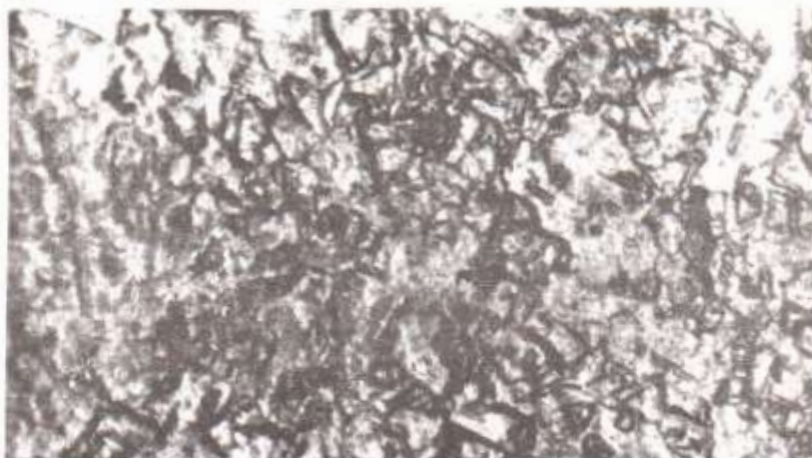

Graphical determination of the activation energy of the recrystallization process.

before the isothermal annealing which means that the activation energy value for the recrystallization process is a pre-history dependent factor.

Table (1): The chemical composition of the samples in weight %

Cu	Fe	Pb	P
balance	0.015	0.005	0.007

Fig. 7: Partially recrystallized pure copper annealed at 350°C/10 min.

Fig. 8: Complete recrystallized pure copper annealed at 350 C/2 hrs.

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