



## STUDY ON THE PRECIPITATION KINETICS OF ORDERED PHASE IN Ni-10 at% Cr ALLOY

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### Abstract:

*The precipitation phenomenon of Ni<sub>3</sub>Cr phase in Ni-10 at% Cr alloy was studied through the isothermal annealing at different high temperatures. It was found that a metastable microstructure is formed during isothermal annealing at 850 °C. The precipitation of ordered Ni<sub>3</sub>Cr phase was obtained during isothermal annealing at 950 and 975 °C. Both processes increase the microstress in the matrix and consequently seemed to impose a heavily pinning action on the magnetic domain walls. The precipitation process was found to be controlled by a second order reaction, activated by an energy of 1.3 eV.*

**Keywords:** Ni-10 At% Cr Alloy; Magnetic Properties; Precipitation Phenomenon; Ordered Phase.

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### 1. Introduction

Significant progress in the study of homogeneous second phase precipitation in alloys has been achieved in the last decade because of the applications of modern serious microscopic techniques to explore the early stages of phase precipitation. It has been established [1-3] that the later stages of precipitation in binary alloys can be described in terms of a simple picture as microstructural evolution. The real materials may not be always homogenous because various types of lattice defects like vacancies, dislocations and domain boundaries might be involved. The precipitation of second phase in alloys was found to be enhanced with the existence of these lattice defects in alloy matrix [3]. The precipitation of second phase in a matrix produces structural changes and the new phase introduces elastic strain, which changes the kinetics of precipitation [4]. The grain boundary precipitation was accelerated by increasing temperature above 800 °C. It was developed very fast and can be formed at very short time [4]. Grain boundary precipitation also causes embrittlement of the alloy. The rate controlling processes for this phenomenon are the volume and the grain boundary diffusion of chromium in the alloy matrix [5]. Accordingly, controversial results on the behavior of the kinetics of precipitation were reported [6]. It is established that the electrical and magnetic properties are very sensitive to structural changes due to the grain-boundary precipitation [7-9]. The precipitation phenomena of chromium in nickel is caused by isothermal annealing at high temperature [10].

From the above consideration, the aim of the present work is to study the precipitation kinetics of the ordered phase in Ni-10 at % Cr alloy using magnetic measurements.

## 2. Experimental Work

The test material, Ni-10 at% Cr alloy, was prepared in the Institute for Ferrous Metallurgy, Moscow, from high-purity Ni and Cr by induction melting followed by a suitable homogenization at 1200 °C under a helium atmosphere for 24 hours then slowly cooled to room temperature.

The material was shaped by extrusion into rods of 3 mm diameter followed by swaging at room temperature to wires of 1 mm diameter. The sample was introduced as the core of the magnetization coil and the cathode-ray technique “digital storage oscilloscope” was employed to obtain room temperature (B-H) curves at different magnetization field.

The samples were heated at 800 °C for 3 hours and slowly cooled, to remove the effect of cold work. These samples were used to study the precipitation kinetics of the ordered phase Ni<sub>3</sub>Cr using magnetic measurements.

In the present work, the magnetic normal permeability,  $\mu$ , was determined from the division of the magnetic induction, B, and the magnetic field, H. The  $\mu_{\max} = (B / H)_{\max}$ , was obtained from the maximum value of the peak ( $\mu$ -H) curves, which characterizes the magnetization of both reversible and irreversible domain wall motion.

## 3. Results and Discussion

Sets of isothermal annealing curves were obtained at different temperatures: a) 850, b) 950 and c) 975 °C, showing the variation of magnetic permeability  $\mu = (B/H)$  with magnetic field (H) (Fig. 1). Each set of curves were characterized by pronounced peaks representing maximum magnetic permeability ( $\mu_{\max}$ ). The dependence of the maximum magnetic permeability ( $\mu_{\max}$ ) on the annealing time ( $t_a$ ) is illustrated in Fig. 2. The present isothermal annealing results can be reported in normalized form of maximum magnetic permeability by using the relation:

$$K_{\text{norm}} = \{[\mu_{\max}(t_a) - \mu_{\max}(i)] / [\mu_{\max}(f) - \mu_{\max}(i)]\} = \Delta\mu_{\max}(t_a) / \Delta\mu_{\max}(0)$$

Where  $\mu_{\max}(i)$  is the initial value of maximum magnetic permeability of cold-worked sample before annealing,  $\mu_{\max}(t_a)$  and  $\mu_{\max}(f)$  are the values after annealing at time ( $t_a$ ), and the value after final isothermal annealing for time ( $t_a$ ).

It is advised that this section be written in past tense. It is a good idea to rely on charts, graphs, and tables to present the information. This way, the author is not tempted to discuss any conclusions derived from the study. The charts, graphs, and table should be clearly labeled and should include captions that outline the results without drawing any conclusions. A description of statistical tests as it relates to the results should be included.

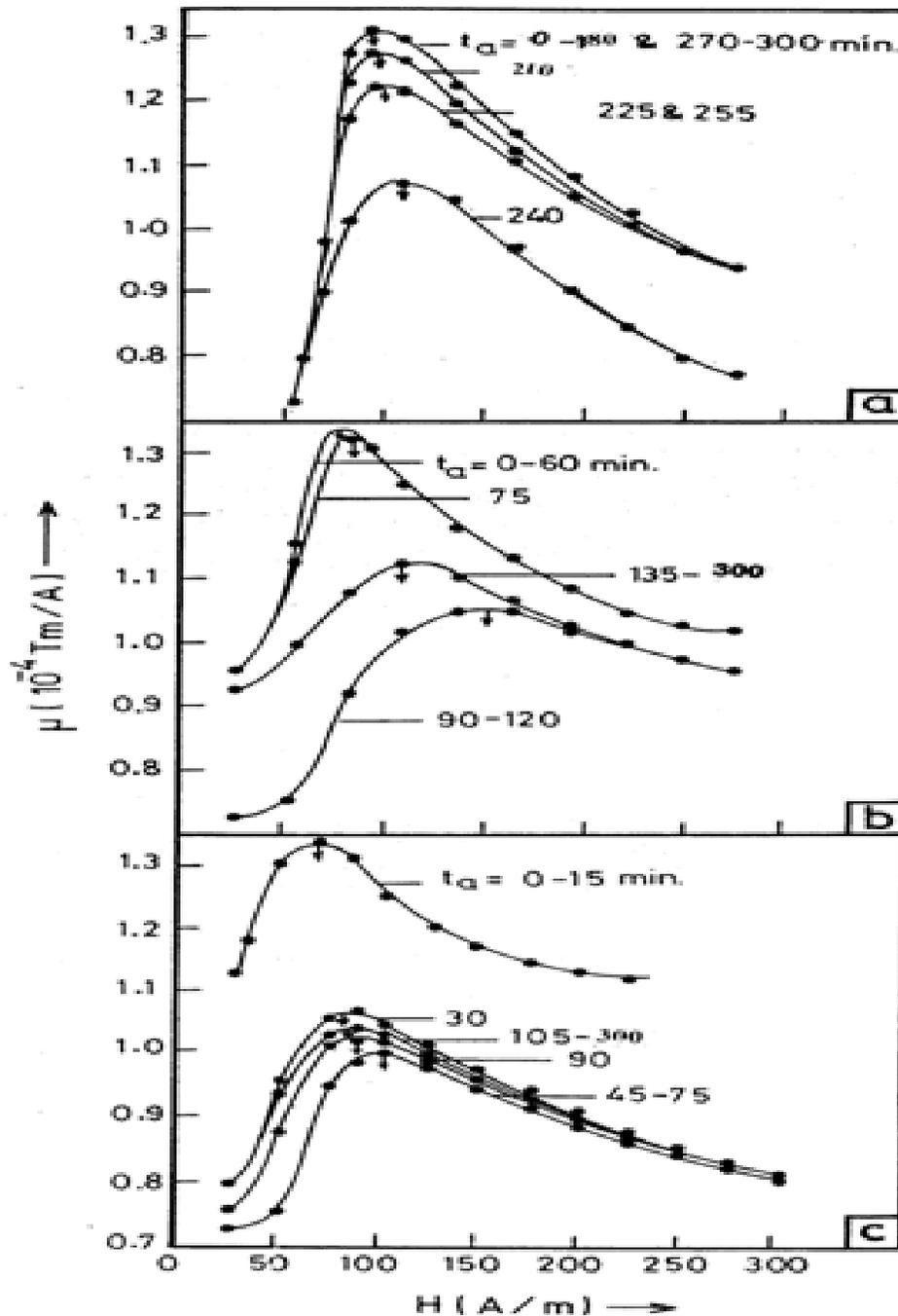


Figure 1: Effect of isothermal annealing on the dependence of the magnetic permeability ( $\mu$ ) on the magnetic field ( $H$ ) of pre-annealed samples at different annealing temperatures: a) 850 , b) 950 and c) 975 °C.

Figure 3 shows the behavior of change in normalized maximum magnetic permeability ( $K_{\text{norm}}$ ) with the annealing time ( $t_a$ ) for different initially pre-annealed samples at different temperatures. The general behavior noticed in the present work is a decrease in  $K_{\text{norm}}$  followed by an increase with the annealing time ( $t_a$ ).

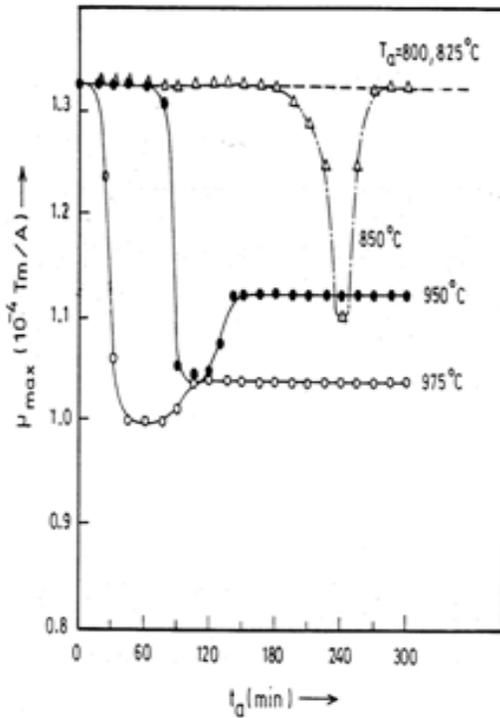


Figure 2: Effect of annealing time on the maximum magnetic permeability of pre-annealed samples of Ni-10 at % Cr alloy at different temperatures.

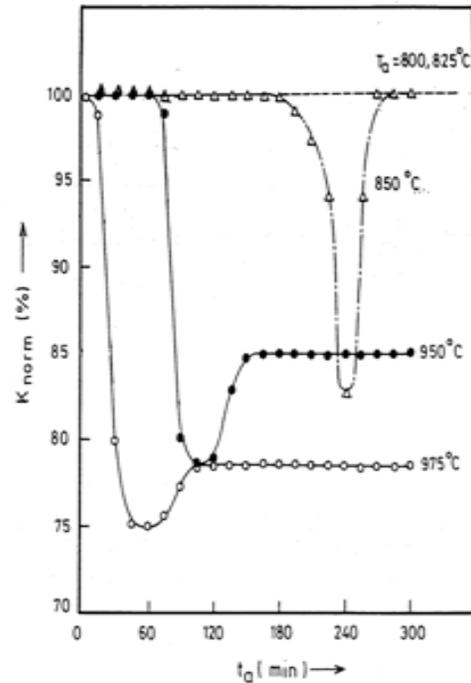


Figure 3: Normalized change of maximum magnetic permeability [ $K_{norm} = \mu_{max}(t_a) / \mu_{max}(f)$ ] during isothermal annealing at different temperatures for pre-annealed samples.

The annealing time to reach the minimum ( $t_{min}$ ) was found to decrease with increasing the pre-annealing temperature. The activation energy of the precipitation process is obtained from the equivalent annealing temperature (Figure 4). By using Meechan and Brinkmann's method [11], it was found 1.3 eV (see Figure 5). The order of precipitation [12] was found as a second order reaction (see Figure 6).

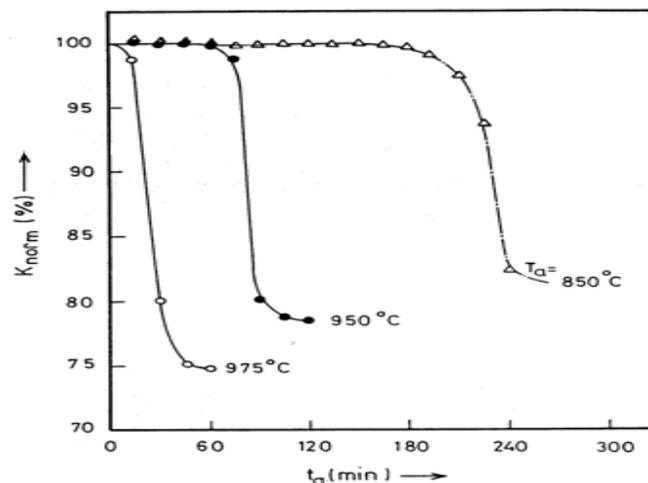


Figure 4: Isothermal annealing of the maximum magnetic permeability of Ni-10 at% Cr alloy at different temperatures for pre-annealed samples.

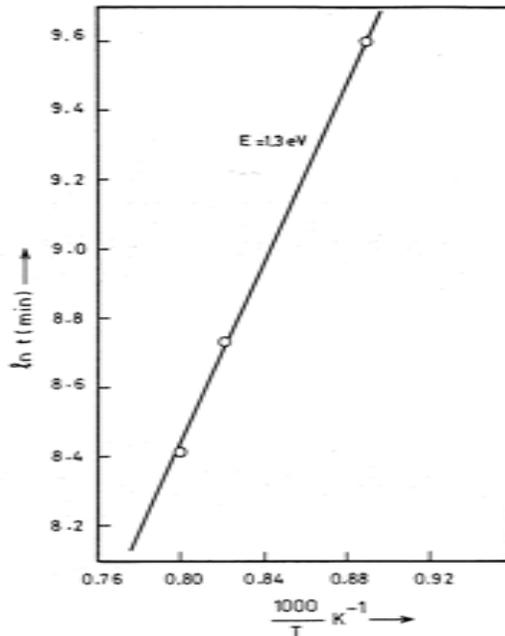


Figure 5: Determination of activation energy using Meehan and Brinkmann's method [11].

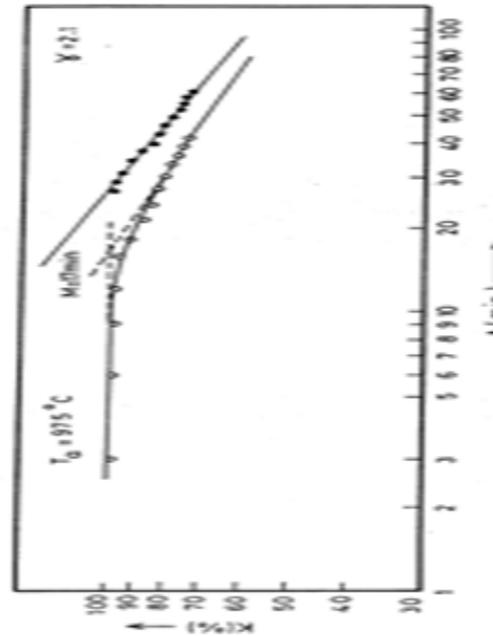


Figure 6: Determination the order of reaction using Ceresara et al. method [12].

The behavior of isothermal annealing at 850, 950 and 975 °C could be explained by two processes which may occur together at the beginning of isothermal annealing namely [13, 14]:

- 1) Formation of a thermally metastable microstructure, and
- 2) Precipitation of ordered Ni<sub>3</sub>Cr phase.

Both processes increase the microstress in the matrix and consequently seemed to impose a heavily pinning action on the magnetic domain walls, preventing them from normal detachment from any fixation points and leak out the magnetic pressure exerted by the magnetic field on domain walls [15, 16]. This effect tends to decrease the maximum magnetic permeability. The dissociation of a metastable microstructure after a relative short heating time during the isothermal annealing at high temperatures ( $T_a > 850$  °C), decreases the internal stress in the alloy matrix, and consequently increases the maximum magnetic permeability [16].

The value of activation energy (1.3 eV) obtained in the present work is in a good agreement with the value of the mean formation energy of thermal vacancies in Ni-alloys [17, 18].

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