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A STUDY OF PHASE TRANSFORMATIONS IN ULTRARAPIDLY QUENCHED ALUMINIUM-IRON ALLOY

E. Babić, R. Krsnik, B. Leontić and I. Zorić
Institute of Physics of the University of Zagreb, Yugoslavia

Introduction

It is not possible to follow the process of formation of solid solutions. One can obtain some information about this process by investigating kinetics and morphology of decomposition of solid solutions. Among numerous methods, that of annealing the samples is one of the best because it enables us to follow the process in optimum time intervals. An easily measurable physical property which strongly depends on these processes is the electrical resistance. Because of that we have chosen resistometric analysis of both kinetics and morphology of phase transition, during the annealing process. The results are compared with those obtained by other authors who used different methods (X-ray measurements, microhardness, electron microscopy).

Experimental procedure

The samples obtained by splat cooling (1) and by the use of a rotating mill device (2) contained iron concentrations between 0.4 and 1.88 at%. For our measurements we selected samples, with surface ranging from 4 to 100 mm^2 and thickness of 10 to 30 μm. Each sample had previously undergone residual resistance measurements. The ratio

$$R = \frac{R_{4.2}}{R_{273} - R_{4.2}}$$

may be considered as a measure of "quality of quenching" of a sample with a given iron concentration (3). The samples were subjected to isochronous annealing starting at room temperature and ending at 420°C. This annealing took place in oil baths whose temperatures increased in steps of about 20°C. The annealing at each temperature lasted for a period of 10 minutes, at the end of which the sample was immediately placed in liquid nitrogen and its resistance measured

by the usual method of compensation. The activation energy for the process of phase transformation was measured by the "ratio of slopes" method.

Results and discussion

Typical behaviour of electrical resistance changes for three samples of two different Fe concentrations as a function of annealing temperature is shown by the curves in Fig.1.

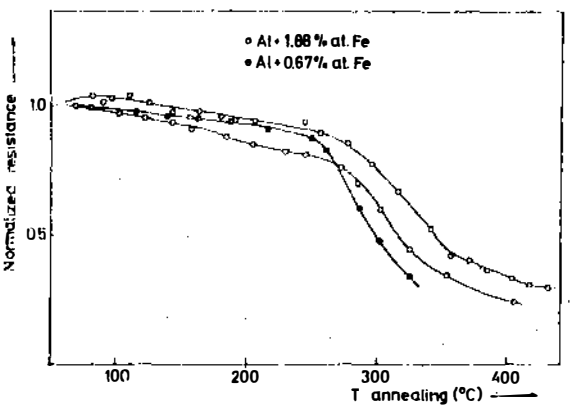

FIG. 1

Electrical resistivity changes for three samples with various Fe concentrations

We see that at higher temperatures of annealing a large change of electrical resistance is observed. Such behaviour starts at temperatures slightly above 200°C and extends up to 350°C. At the end of this region electrical resistance is down on 25% of its original value for all samples. Such a behaviour is typical for phase transition. During further annealing at temperatures higher than 350°C the process of recovery continues as evidenced by a slow decrease in resistance. Limitations of experimental technique prevented us from following the process beyond 420°C. From Fig.2 we can conclude two important things: a) The rate of phase transformation is largest in temperature region 250-300°C, and b) for annealing temperatures of about 400°C high resistance ratio and behaviour of the curve $\frac{R_{295}}{R_{77}}(T_{ann})$ show that the transition is over. That is, the system

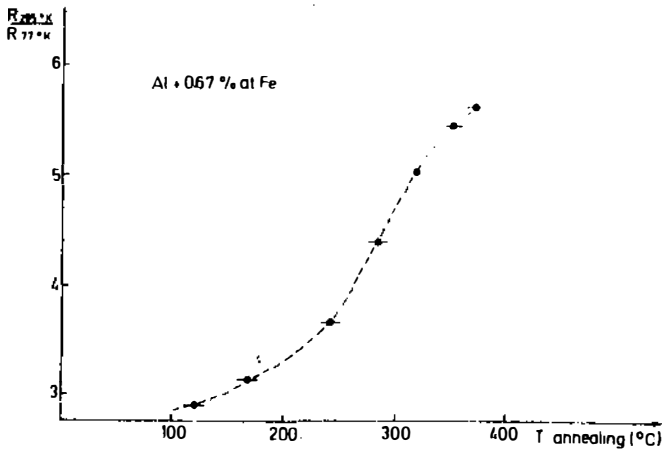

FIG. 2

The ratio $\frac{R_{297}}{R_{77}}$ versus temperature of the isochronous annealing (Time interval of the annealing is 10 min)

consists of pure Al and Al_6Fe, in accordance with (4) and (5).

At temperatures where the phase change takes place, activation energies were measured for the process. From our curves of isochronal annealing, X-ray diffraction (4) and electron microscopy measurements (5) we believe that there is a single activated process in the temperature interval of the phase transformation. Because of that we used a "ratio of slopes" method for determination of the activation energies of the process. For each sample the activation energy was measured at several temperatures between 250 and 300°C. A mean value of (1.2 ± 0.3) eV has been derived. It turns out that this activation energy is substantially independent of iron concentration in this concentration interval, but shows a tendency of slow increase with temperature. However we have not been able to establish a clear functional dependence an temperature owing to considerable errors which this method entails.

In order to obtain more precise results on the kinetics of decomposition of these solid solutions we intend to apply isothermal annealing and the combination of

isochronal and isothermal annealing in the temperature region of the phase transition. Also we intend to extend the annealing to higher temperatures up to the formation of Al_3Fe phase. The whole experiments should be done once more with magnetic susceptibility measurements, to see the fractional composition of the sample (Al+Fe, Al_6Fe and Al_3Fe) at any moment during annealing. This work is beeing pursued.

Conclusion

Our measurements show definite existence of a phase transition at annealing temperatures around 300°C. The annealing temperature at which this phase change takes place does not depend significantly on iron concentration within concentration interval studied. The fact that no phase change occurrs below 250°C as well as the large temperature interval over which this transition proceeds indicates a relatively high stability of the solid solution in the interval of iron concentration studied. These measurements are consistent with X-ray (4) and electron microscopy measurements (5).

Acknowledgements

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References

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   Some of earlier electron microscopy results on annealed samples are published in Metallography 3 (1970) 79

DISCUSSION

H. Warlimont : Resistivity changes can be produced by numerous processes (annealing out of defects, nucleation, growth, coarsening etc which are associated with different activation energies. The association of a resistivity change with a specific process can, therefore, only be unequivocally established by determining the nature of this process by some other means of observation. Which of the processes associated with the precipitation of Al_6Fe is giving rise to the activation energy which you have determined from your resistivity measurements?

R. Krsnik : From electron micrographs it can be seen that the rapid particle coarsening takes place between 200 and 400°C, together with a drastic change in the type of the precipitation patterns (Reference: D. Kunstelj and A. Bonefačić, Metallography 3, (1970) 79. This work is done in the samples produced by the use of the same splat cooling equipment.

B. Leontić : If we had indeed two distinct processes we would generally get two steps in the curve. The fact that we get only one seems to indicate that we are either dealing with only one process or that we have indeed more processes that by accident occur at the same temperature. In any case our interpretation is put forward in conjunction with the results of our colleagues working with X-ray and electron microscopy.

T. Ungar : There are many cases when resistivity measurements give perhaps the only means for following a phase transition. This is the case especially at the very beginning of a process (e.g. in Al MgSi) when the amount of the new phase is too small to be detectable by diffraction means.

R. Roberge : Only a short comment on the determination of apparent activation energies. With liquid-quenched Al-40 wt% Ag studied over the same temperature range using electrical resistance and high-angle X-ray the following was obtained: electrical resistance 20 $\pm$ 2 Kcal/mole high-angle X-ray 27 $\pm$ 2 Kcal/mole. There is no way I know to reconciliate the two estimates.

Đ. Lazarević : Suggestion: Better, probably to combine the isochronal and isothermal data (Mechan. Brinkmann, Phys. Rev. 103, (1956) 1193.) This will give a straight line (if there is a unique annealing process), which slope is used for activation energy determination. The method presented in the paper implies a unique annealing process.

R. Krsnik : It is intended to do that in further work.

R.W. Cahn : We now have so many reliable techniques for identifying physical processes responsible for changes in microscopic processes in metals+alloys, that perhaps we should declare a moratorium on the determination and interpretation of activation energies.

A. Guinier : Studies of simple and well known systems (Al-Ag, Al-Zn) have shown the difficulty to reconcile resistivity measurements and structural X-ray or electromicroscopic investigation.