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THERMAL INVESTIGATIONS OF MAGNETICALLY HARD Fe-Cr-Co ALLOY

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Differential thermal analysis and differential scanning calorimetry were applied to examine magnetically hard Fe-Cr-Co alloy. The spinodal decomposition temperature of this materials was established.

The magnetically hard Fe-Cr-Co alloys were first described in 1971 [1]. Their magnetic properties were found to be dependent on the chemical composition (Fe, Cr, Co and alloy additives) and the heat treatment technology [2-5]. The mechanism of the magnetic hardening of these materials was explained in terms of the α_1 and α_2 structures formed (α_1 = ferromagnetic phase rich in Fe and Co, α_2 = weakly magnetic phase rich in Cr) as a result of the spinodal decomposition.

The range of the magnetic properties was a function of the volume contribution of the phases, their respective dispersions, the shape and size of the particles of the α_1 phase, and the chemical compositions of both phases. The elements of the microstructure geometry described above were dependent on the heat treatment technology.

The typical heat treatment of Fe-Cr-Co alloys involves solutioning from the temperature at which the alloys have the monophase α structure and ageing, during which spinodal decomposition occurs.

Thermal investigations permit establishment of the optimization temperatures and the time of ageing of alloys [6].

The alloy containing 25% Cr and 12% Co, after solutioning at 1473 K for 1 hour and cooling in water, was tested by different thermal analysis (DTA). The alloy was made in the Institute of Material Engineering of the Technical University in Warsaw.

The investigations were performed with a 1090 Du-Pont thermal analyzer. The cylindrically shaped samples were placed in the alumina

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crucible on the separate Pt/13Rh-Pt thermocouple. The measurements were made in air with α-Al_2O_3 as the reference material.

During the dynamic cooling process at a rate of 2 deg/min in the temperature range 1473-573 K, the thermal curve $\Delta T = f(T)$ was recorded. A large exothermic effect was observed in this curve (Fig. 1); the starting temperature $T_S = 913$ K and the temperature range of spinodal decomposition (913-793 K) were established.

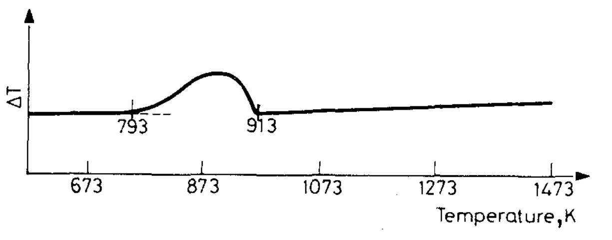


Fig.1 DTA curve of the solutioned Fe - Cr25 - Co12 alloy cooled at 2 deg/min

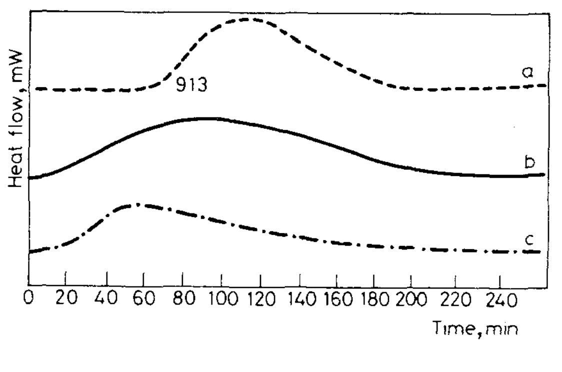
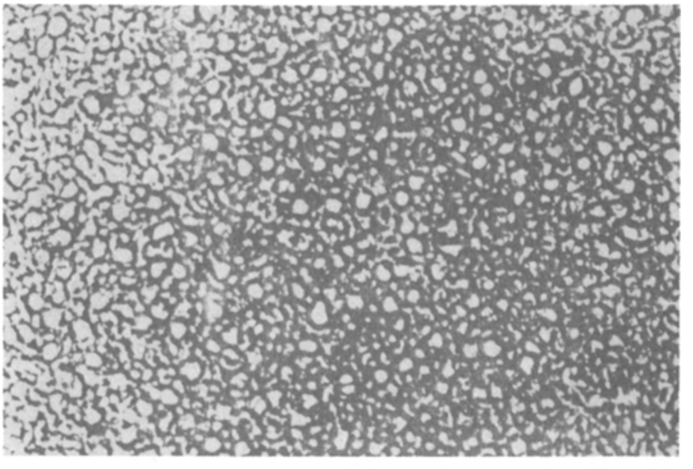


Fig. 2 DSC curve of the solutioned Fe - Cr25 - Co12 alloy aged at a) - 918 K, b) - 913 K, c) - 903 K and cooled at 0.5 deg/min to 793 K

The material was tested under conditions of high-temperature ageing at the following temperatures: higher than T_S (918 K), equal to T_S (913 K), and lower than T_S (903 K) for 1 hour and cooled at a rate of 0.5 deg/min to 573 K (Fig. 2). The heat flow during spinodal decomposition was small and the differential scanning calorimeter method (DSC) was used. This method is more sensitive for the heat flow and the determination of the precise temperature than the DTA method. The sample was placed in the nickel crucible, which was previously annealed. The reference material was α-Al_2O_3.

The exothermic effect in the thermal curve was not observed during annealing for 1 hour at the temperature above T_S. Spinodal decomposition did not take place. The exothermic effect induced by the spinodal decomposition was recorded only at 913 K when the sample was slowly cooled (Fig. 2a). After this thermal treatment, the structure of the alloy contained

a)



b)

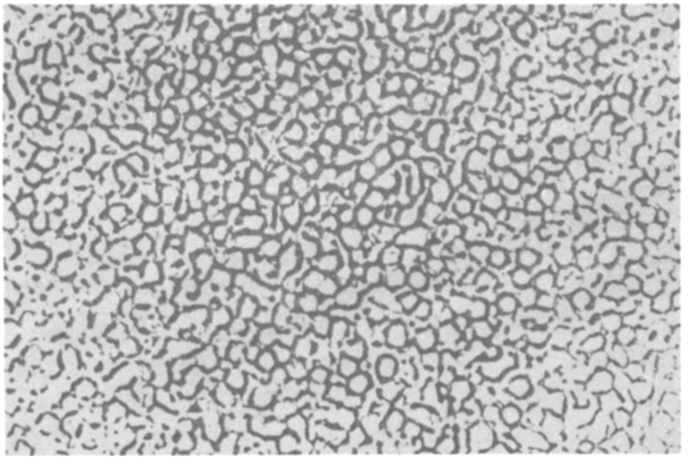


Fig. 3 Fe - Cr25 - Co12 alloy microstructure after high-temperature ageing at a) - 918 K, b) - 913 K. TEM 100 000x

small particles of the α_1 and α_2 phases. This fact was confirmed by microscopic observations (Fig. 3a).

During the ageing of the material at temperatures equal to T_S and lower than T_S, in the entire range of temperature measurements the exothermic effect of spinoidal decomposition was observed (Fig. 2b, c). Microstructural observations are shown in Fig. 3b.

Conclusion

The thermal investigation of the Fe - Cr25 - Co12 alloy revealed the starting temperature of the high-temperature ageing. This process should start from a temperature not higher than T_S, and the time for which the material is left in the spinodal decomposition temperature range should be as long as possible for the growth of particles of the α_1 and α_2 phases. The thermal measurements confirmed that small oscillations of the ageing temperature ($\pm$10 deg) produced a diversification of the microstructure geometry and a considerable spread in the magnetic properties of the material.

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Zusammenfassung — Mittels DTA und DSC wurden hartmagnetische Fe-Cr-Co Legierungen untersucht. Es wurde auf die spinodale Entmischungstemperatur dieser Materialien aufmerksam gemacht.