

Coercivity dependence of a $\text{Sm}_2(\text{Co}, \text{Cu}, \text{Fe}, \text{Zr})_{17}$ type alloy on magnetic processing procedure

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The results of an investigation into the effects of the magnetic processing procedure on the intrinsic coercivity of a $\text{Sm}(\text{Co}_{0.673}\text{Cu}_{0.080}\text{Fe}_{0.222}\text{Zr}_{0.025})_{8.92}$ (2:17-type) alloy are reported. Two basic types of magnet were investigated: polymer-bonded fine powder magnets and cast (solid) magnets. The fine particles which were processed into polymer-bonded magnets were prepared by two different methods; i.e. by milling or by a hydrogen treatment. The cast magnets were manufactured from selected parts of solidified ingots exhibiting preferred orientation and along directions parallel to the preferred orientation of magnetization. Magnetic properties and Vickers microhardness measurements on the solid solution treated (1170°C) and isothermally aged (800°C) samples, revealed that there was a clear similarity between the variations of the intrinsic coercivity and microhardness values versus ageing period. This suggests a coercivity mechanism for the present 2:17-type alloy which is predominantly controlled by general domain wall pinning by a critical dispersion of coherent precipitates. Certain aspects of the intrinsic coercivity against ageing time variations of the variously processed magnets as well as the corresponding microhardness variations have been attributed to a partial conversion of coherent precipitates to semi- or in-coherent particles during processing.

1. Introduction

The $\text{Sm}_2(\text{Co}, \text{Cu}, \text{Fe}, \text{Zr})_{17}$ type magnetic materials have become the focal point of research and development in the field of the rare earth-transition metal 2:17-type ($\text{RE}_2\text{TM}_{17}$) compounds (see, for example [1–11]). A popular composition range for these alloys can be characterized by the general formula $\text{Sm}(\text{Co}_{\text{bal.}}\text{Cu}_{0.08}\text{Fe}_{0.22}\text{Zr}_{0.01-0.03})_Z$, where $7 < Z < 9$ [1–3, 6–9, 11]. The role of heat treatments, in particular the solid solution treatment (SST) process, has been found to be critically important in developing the hard magnetic properties [3, 12]. An optimum SST temperature has been established within the range 1160 to 1180°C for the $\text{Sm}(\text{Co}_{\text{bal.}}\text{Cu}_{0.08}\text{Fe}_{0.22}\text{Zr}_{0.01-0.03})_Z$ compounds with $7 < Z < 9$. The influences of the subsequent isothermal or step-ageing processes, on the intrinsic coercivity (iH_c) values, have been described in many different

publications (see, for example [1–3, 6–9]). An isothermal ageing temperature of 800°C has generally been adopted as the optimum temperature for these compounds [1, 3, 7, 12].

The present paper reports some observations on the influence of the precise procedure by which the magnets are produced (after an identified heat treatment) on the iH_c values. The interpretation of the magnetic results has been supported by establishing a correlation between the magnetic and mechanical hardness of the specimens. We have previously obtained a convincing correlation between the microhardness and the iH_c values for some precipitation-hardened 2:17-type magnetic materials [13] and in this paper the microhardness variations are used to provide further evidence of the nature of the precipitates and hence the nature of iH_c variations with time.

2. Experimental procedure

Specimens of a commercial alloy with composition given by $\text{Sm}(\text{Co}_{0.673}\text{Cu}_{0.080}\text{Fe}_{0.222}\text{Zr}_{0.025})_{8.92}$ were solid solution treated (SS treated), in evacuated silica tubes under 0.5 atm purified argon, at 1170°C for 4 h and were then quenched into water in the argon atmosphere. The SS treated samples were subsequently isothermally aged at 800°C for 1 to 50 h.

The large number of polymer-bonded cylindrical test magnets (prepared after each stage of the isothermal ageing treatment) were made of magnetically aligned particles of:

1. milled and sieved samples with an average particle size of $40\ \mu\text{m}$; and
2. of hydrogen treated (HT) powder with an average particle size of $100\ \mu\text{m}$.

The cast (solid) test magnets were produced from directionally preferred solidified specimens. These specimens were cut along directions parallel to the preferred orientation of magnetization, carefully ground into the cylindrical test magnets and then heat treated in an identical manner to that described above. The isothermal ageing treatment of one of the cast magnets was, however, carried out in a magnetic field in an attempt to investigate the effects of such a treatment on the iH_c values.

A part of each of the specimens, used for the study of the isothermal ageing behaviour of the polymer-bonded magnets, was utilized for the hardness determinations, thus ensuring the strict comparability of the magnetic and hardness measurements. However, to minimize any effects of surface preparation on the hardness values, the SS treated samples were ground, prior to the ageing treatment, to a 1200 grit finish. The crystal damage associated with the grinding process was, therefore, believed to be recovered during the ageing process. The aged samples were mounted in hard bakelite in order to obtain highly parallel surfaces and were then finally mechanically polished with $\frac{1}{4}\ \mu\text{m}$ diamond paste. The hardness tests were performed on unetched samples using a "Leitz" microhardness tester. An average value was obtained from ten measurements made on each sample and uniform readings were obtained across all the specimens which were typically $\frac{1}{2}$ cm diameter.

3. Results

The variations of the iH_c values with isothermal

ageing time of a series of polymer-bonded magnets (made from milled and HT materials) and a cast magnet are shown in Fig. 1. The curves of iH_c against ageing period of all three types of magnet exhibited maxima after 38 h ageing; i.e. overageing occurred after this time interval in all three types of magnet. The highest values of iH_c were observed for the cast magnet followed by the magnets produced from the HT material, with the magnets made from the milled powder exhibiting the lowest values of iH_c . Initially the rate of increase of iH_c was highest in the cast magnet and lowest in the milled specimens, with the magnets produced from the HT material exhibiting an intermediate rate. The rate of fall in the iH_c values of the cast magnet, after the maximum, was considerably lower compared with the sharp reduction of iH_c in the bonded magnets which was particularly pronounced in the case of the magnets made from the milled material.

The variation of the microhardness (Vickers hardness number, VHN) with ageing period is shown in Fig. 2 and this also displays a maximum at 38 h ageing. Thus there is a close similarity between the variation of VHN and iH_c (either bonded or cast magnets) with the ageing period. This can easily be observed if curves of Fig. 1 are compared with that of Fig. 2. However, the sharp fall of VHN, after the maximum, is more closely followed by the iH_c curves of the bonded magnets.

Fig. 3 depicts the comparison between the variations of the iH_c against ageing time of the cast magnet with another identical cast specimen aged in a magnetic field. For all but the largest ageing period, the values of iH_c were significantly higher in the magnetically treated magnet than in the non-magnetically treated counterpart, however, there is tentative evidence that the rate of iH_c fall, past the maximum, was higher in the former magnet than in the latter.

4. Discussion

The close similarity in the forms of Figs. 1, 2 and 3, clearly illustrates that the magnetic and mechanical "age-hardening" phenomena occur simultaneously during the ageing treatment. It is known that the coercivity of the precipitation-hardened magnetic alloys is predominantly controlled by the interaction of moving domain walls and precipitates (see, for example [14, 15]). The hinderances to domain wall motions in these alloys which determine their magnetic hardness,

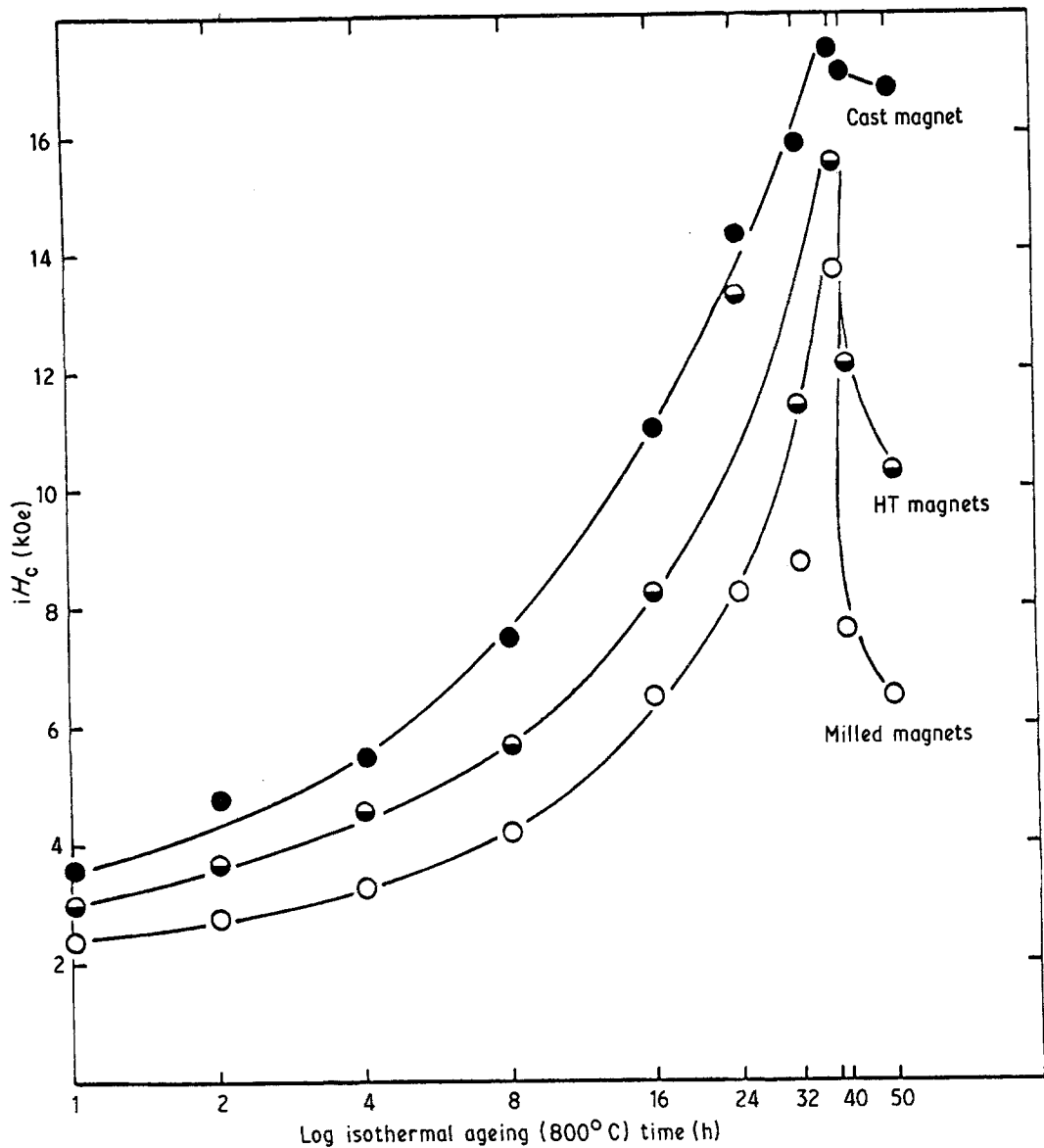


Figure 1 Variation of iH_c values with isothermal ageing (800°C) period for cast, HT and milled magnets.

may be due to microstrains associated with coherent precipitates and the more direct interaction of the precipitates with the domain walls. Similarly, the mechanical hardness of age-hardened alloys has been ascribed to the interaction of dislocations and precipitates (see, for example [16, 17]). The obstacles which impede the motion of dislocations have been related to microstrains around coherent precipitates and the more direct interaction with the precipitates themselves. The present results which show a close correlation between the variation in magnetic hardness (iH_c) and mechanical hardness with ageing time (Figs. 1

and 2), clearly indicate that domain wall and dislocation pinning in this alloy occur together so that overageing occurs after an identical ageing period. The coercivity of the present 2:17-type material may now safely be related to a general pinning of the domain walls by a critical dispersion of coherent precipitates. This is, in fact, confirmation of previous investigations [1, 3, 6, 10, 12] by a new method of microhardness assessment.

There are two interesting features in the plots of iH_c against time for the cast magnet, when compared with the bonded specimens:

1. the higher values of iH_c for the cast magnet;

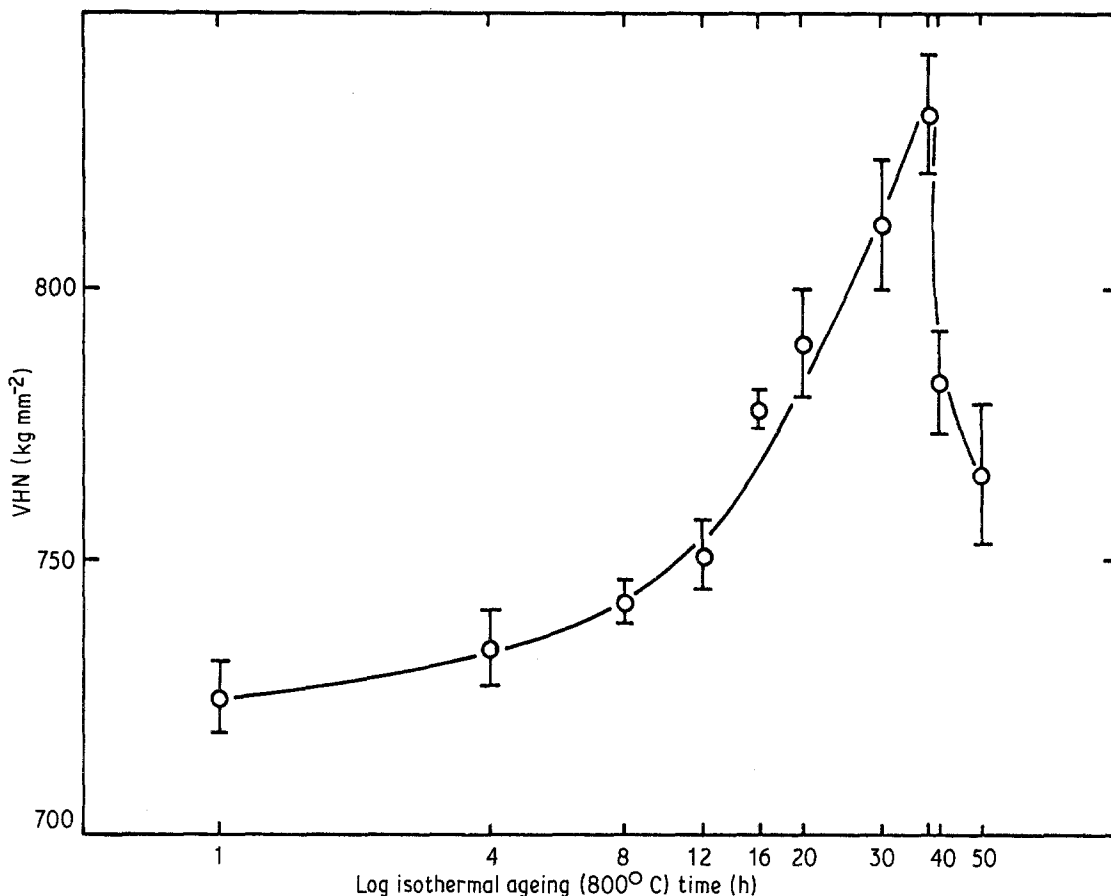


Figure 2 Vickers hardness values plotted against isothermal ageing (800°C) period.

and

2. the much lower rate of its decrease after the maximum.

Both these effects may be related to the lack of any mechanical comminution associated with the production of the cast magnet. Thus, in the bonded specimens, it could be argued that the mechanical comminution causes the conversion of those coherent precipitates which are in a very unstable state into semi- or in-coherent particles. This is obviously more pronounced after the iH_c maximum, since the number of unstable coherent precipitates which are prone to incoherency will be increasingly dominant with increasing ageing time. In addition, the crystal defects produced by decrepitation, might also provide easy nucleation sites for reverse domain walls and this would be consistent with the lower values of iH_c up to the maximum and the higher rate of decrease after the maximum, in the bonded magnets; this is, however, unlikely to be the dominant mechanism in the precipitation-hardened magnetic alloys. The

higher iH_c values of the HT magnets compared with the magnets made from the milled material, may be attributed to significantly lower mechanical deformation associated with the production of the HT particles [18].

It should be noted that there is a lack of any marked dependency of iH_c on the particle size (comparing cast magnets with HT specimens with average particle size of $\sim 100\mu\text{m}$ and milled samples with average particle size of $\sim 40\mu\text{m}$) and this can be considered as further evidence for the general domain wall pinning mechanism in the present material.

One of the significant features of the plot of VHN against time is its sharp reduction after the critical ageing period which is in accordance with the rapid falls of iH_c of the bonded samples. This sharp decrease of the VHN is believed to be not only due to the overageing phenomenon but also to the plastic deformation associated with the hardness measurements; the latter is thought to convert critically unstable coherent precipitates

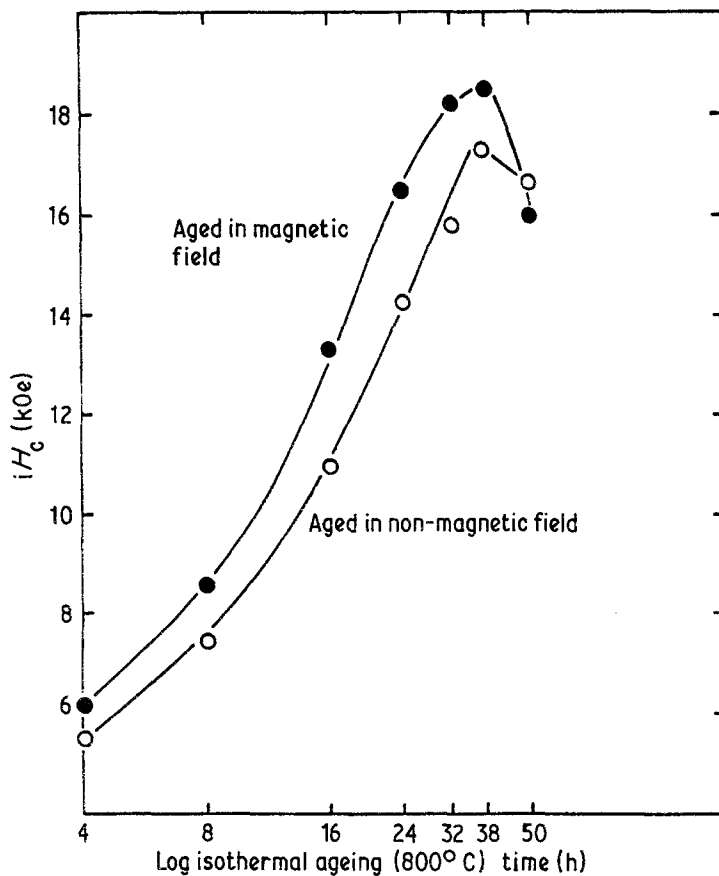


Figure 3 Variation of iH_c values of cast magnets with isothermal ageing (800°C) period.

to equilibrium semi- or in-coherent particles, i.e. a similar process to that which has been proposed earlier to explain the rapid fall in iH_c of the bonded magnets after the maxima.

In the magnetically treated cast magnet, the higher values of iH_c up to and including the maximum, and the more rapid fall past the maximum, when compared with identical counterpart aged in a non-magnetic environment, might both be related to the influence of the applied magnetic field. It could be argued that the magnetic field effects the nucleation, growth, shape and array of the coherent precipitates, in the period up to the iH_c maximum, and facilitates the change of the critically unstable coherent precipitates into the stable incoherent particles, after the maximum. A detailed study of the effects of applied magnetic fields on the iH_c values is the subject of further studies.

References

1. T. YONEYAMA, S. TOMIZAWA, T. HORI and T. OJIMA, Proceedings of the 3rd International Workshop on RE-Co P.M. and their Applications, Paper No. VII-1 (1978) p. 406.
2. T. OJIMA, S. TOMIZAWA, T. YONEYAMA and T. HORI, *IEEE Trans. Magn.* MAG-13 (1977) 1317.
3. T. SHIMODA, K. KASAI and K. TERAISHI, Proceedings of the 4th International Workshop on RE-Co P.M. and their Applications, Paper No. XI-2 (1979) p. 335.
4. T. SHIMODA, I. OKONOGI, K. KASAI and K. TERAISHI, *IEEE Trans. Magn.* MAG-16 (1980) 991.
5. T. SHIMODA, I. OKONOGI and K. TERAISHI, Proceedings of the 5th International Workshop on RE-Co P.M. and their Applications, Paper No. X-3 (1981) p. 595.
6. R. K. MISHRA, G. THOMAS, T. YONEYAMA, A. FUKUNO and T. OJIMA, *J. Appl. Phys.* 52 (1981) 2517.
7. C. SOCHEN, W. RUN, C. CHENGYI, H. YAOFU and S. GUANGFEI, Proceedings of the 5th International Workshop on RE-Co P.M. and their Applications, Paper No. IX-3 (1981) p. 459.
8. L. RABENBERG, R. K. MISHRA and G. THOMAS, *J. Appl. Phys.* 53 (1982) 2389.
9. T. D. SUN, Proceedings of the 6th International Workshop on RE-Co P.M. and their Applications, Paper No. PIV-7 (1982) p. 433.
10. F. ROTHWART, Y. TAWARA, K. OHASHI, J. FIDLER, P. SKALICKY, R. GRÖSSINGER, H. KIRCHMAYR, S. LIU and K. J. STRNAT, *ibid.*, p. 567.

11. L. RABENBERG, R. K. MISHRA and G. THOMAS, *ibid.*, p. 599.
12. A. KIANVASH and I. R. HARRIS, *J. Less-Common Met.* to be published.
13. *Idem*, *J. Mater. Sci.* to be published.
14. A. MENTH and H. NAGEL, Proceedings of the 2nd International Symposium on Magnetic Anisotropy and Coercivity of RE-TM Alloys, Paper No. 4 (1978) p. 65.
15. J. D. LIVINGSTON, *J. Appl. Phys.* **52** (1981) 2544.
16. R. E. REED-HILL, "Physical Metallurgy Principles" (Van Nostrand, New York, 1970) p. 236.
17. R. E. SMALLMAN, "Modern Physical Metallurgy" (Butterworths, London, 1980) p. 405.
18. A. KIANVASH and I. R. HARRIS, to be published.

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