A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 55 2010 Issue 2

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INVESTIGATIONS OF THERMO-MAGNETIC TREATMENT OF ALNICO 8 ALLOY

BADANIA PROCESU OBRÓBKI CIEPLNO-MAGNETYCZNEJ STOPU ALNICO 8

Studies of the coercivity of Alnico 8 alloy, containing 5.5% Ti, were carried out. The effect of thermo-magnetic treatment parameters on alloy properties was studied. The heat treatment comprised homogenisation at 1250 °C for minimum 20 min, followed by cooling to room temperature in or without magnetic field. After homogenisation, three-step isothermal annealing was performed, holding the sample for 2 h at 610 °C, for 4h at 590 °C, and for 6 h at 570 °C. It was found that the highest coercivity of 132 kA/m was obtained for the heat treatment in magnetic field at 810 °C. At this temperature, the process of magnetic hardening was most efficient. At an optimum temperature of the thermo-magnetic treatment (810 °C), the application of magnetic field during homogenisation is not really of great importance. The heat treatment in magnetic field during homogenisation improves the magnetic properties when the thermo-magnetic annealing is carried out at temperatures too high, but even then the said process cannot provide the properties as good as those that are achieved under optimum heat treatment conditions.

Przeprowadzono badania koercji stopu Alnico 8 zawierającego 5.5% Ti. Określono wpływ parametrów obróbki cieplnej w polu magnetycznym na właściwości magnetyczne badanego materiału. Obróbka cieplna polegała na wyżarzaniu homogenizującym w temperaturze 1250 °C przez min 20 min, z następnym chłodzeniem do temperatury otoczenia w polu magnetycznym lub bez. Dalsza obróbka cieplna obejmowała wyżarzanie w zakresie temperatur 810-850 °C przez 20 min w polu magnetycznym, z następnym chłodzeniem do temperatury otoczenia. Po tym następowało trzystopniowe odpuszczanie w temperaturze 610 °C przez 2 h, 590 °C przez 4h i 570 °C przez 6 h. Stwierdzono, że najwyższą koercję, 132 kA/m, uzyskano dla próbek obrabianych w polu magnetycznym w temp. 810 °C. W tej temperaturze proces utwardzania magnetycznego przebiega najefektywniej. W optymalnej temperaturze obróbki cieplno-magnetycznej (810 ^{circ}C) zastosowanie pola magnetycznego w trakcie homogenizacji nie odgrywa istotnej roli. Pole magnetyczne przy homogenizacji polepsza właściwości przy obróbce cieplno-magnetycznej w zbyt wysokich temperaturach, ale procesy te nie pozwalają na uzyskanie parametrów magnesu obrabianego w optymalnych warunkach.

1. Introduction

Magnets from the Alni and Alnico families were developed by Mishima in early 30-ties of the past century. The alloy of a roughly Fe_2NiAl formula developed by Mishima has soon become the starting product for all other alloys included in this group of magnetic materials. The said magnets are characterised by high remanence, low coercivity, low magnetic energy, and high temperature stability. Depending on alloy grade, the Curie point is 810-860 °C at the service temperature of 450-550 °C [1]. Despite the fact that Alnico magnets have a long, for over seventy years old, history, from the technical point of view they are still considered a very important family of the magnetically hard materials.

Even when melted for the first time, the Alni alloys were modified with various alloying elements (Co, Ti, Cu, S, Nb), while their fabrication techniques were subject to permanent improvements (treatment in magnetic field and various methods used to produce a columnar structure). All these efforts have finally resulted in the magnetic energy raised to a level of 72 kJ/m^3 for alloys with columnar grains, heat treated in magnetic field. The Alni and Alnico magnets used nowadays usually contain 0-40%Co, 12-30%Ni, 7-14%Al, 0-8%Ti, 0-6%Cu, and Fe as a rest [2].

Alnico magnets containing above 24% Co are the only group of magnetically hard materials that can be heat treated in external magnetic field. Owing to high cobalt content, considerably raising the temperature of mag-

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netic transformation, it is possible to have the phase distribution process taking place in magnetic field with the resulting formation of anisotropic structure and magnetic hardening effect. The process is of a diffusive character and consists mainly in precipitation of strongly magnetic phase (Fe-Co) which, in the form of oblong microprecipitates elongated in direction of the magnetic field effect, is embedded in a matrix of the weakly magnetic phase (Al-Ni).

In the Fe-Al-Ni-Co-Ti-Cu system at different temperatures, within the range of compositions corresponding to permanent magnets, the following phases exist in an equilibrium state:

- α_1 – ordered solid solution rich in Fe and Co (body centred cubic lattice);
- α_2 ordered solid solution rich in Al and Ni (body centred cubic lattice); α_1 and α_2 are coherent phases;
- γ_1 – disordered solid solution based on γ Fe (face centred cubic lattice).

The solid solution γ_1 occurs only at high temperatures, i.e. at ~850 °C. Below this temperature, at standard cooling rates, it transforms into a body centred cubic phase, different from α_1 and α_2. Taking into account its origin, this phase has been denoted as α_γ.

Figure 1 shows a hypothetical phase equilibrium diagram plotted for Alnico magnets. During standard heat treatment, the structure of an $\alpha_1 + \alpha_2$ type should be produced. To obtain this goal, magnets are homogenised within a range of the α phase formation temperatures and then are cooled at the same rate to avoid, on one hand, the precipitation of γ_1 phase at high temperatures and, on the other, to promote at lower temperatures the formation of α_1 and α_2 phases [3].

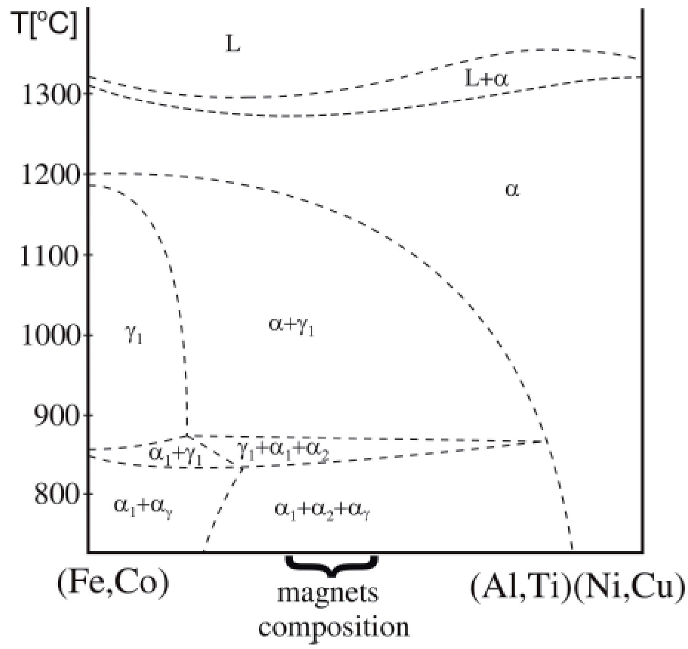

Fig. 1. Scheme of equilibrium phase diagram for Alnico magnets [3]

If cooling is carried out from the temperature too low, i.e. from the $\alpha + \gamma_1$ range, or if the material can undergo a rapid decomposition of the $\alpha + \gamma_1$ phase, e.g. owing to the presence of nuclei of the γ_1 phase capable of further growth, then at room temperature the undesired structure of $\alpha_1 + \alpha_2 + \alpha_\gamma$ will be produced. Figure 2 shows the TTTi diagram for Alnico 400 alloy [3]. The magnet without the crystallisation nuclei of γ_1 phase (curve 1) develops on cooling the beneficial two-phase $\alpha_1 + \alpha_2$ structure, while the same material containing the nuclei of γ_1 phase will develop the undesired structure of $\alpha_1 + \alpha_2 + \alpha_\gamma$ type. Rapid cooling, applied mainly to avoid the formation of γ_1 phase (curve 2), cannot in this particular case give the expected results, because the α_1 phase will have no time to precipitate in a sufficiently large volume. From Figure 1 it follows that there is still another variant of the heat treatment of Alnico magnets that can be carried out. Because of long incubation period of the $\alpha \rightarrow \alpha_2 + \alpha_2$ transformation, at a temperature of approximately ~900 °C, this transformation can be arrested for a time sufficiently short (about 0.5 h) to prevent the precipitation of γ_1 phase, followed by cooling to speed up the decomposition into $\alpha_1 + \alpha_2$ phases.

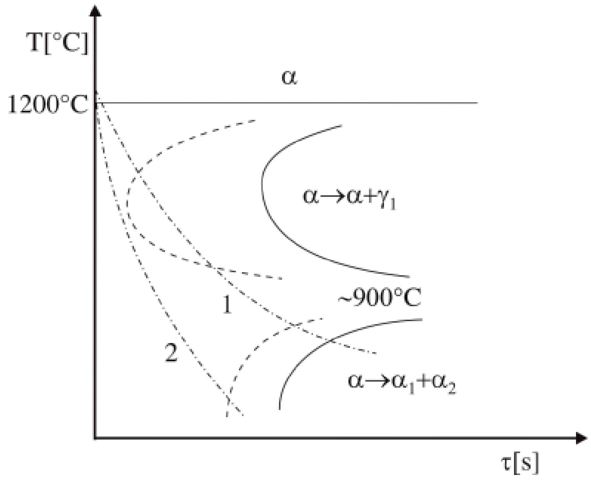

Fig. 2. TTTi diagram for Alnico alloy without crystallization nuclei (onset of the transformation – continuous line) and with crystallization nuclei (onset of the transformation dashed line) [3]

Magnets have optimum magnetic properties when their structure is composed of a mixture of $\alpha_1 + \alpha_2$ phases of the linear dimensions of a few dozen nm.

2. Experimental

Samples for investigations of the heat treatment process were prepared from Alnico 8 alloy (Al – 8.2%; Ni – 14.5%; Co – 36%; Ti – 5.5%; Cu – 3%; Fe – rest) melted in an induction furnace and cast into sand moulds.
The heat treatment of the cast alloys consisted of 20 minute homogenisation at a temperature of 1250 °C, followed by cooling with a rate of 1 – 1.5 °/s in or without a magnetic field, isothermal annealing in magnetic field (thermo-magnetic treatment TMT) at three temperatures 850, 830 or 810 °C, and three-step isothermal annealing carried out for 2 h at a temperature of 610 °C, for 4 h at a temperature of 590 °C, and for 6 h at a temperature of 570 °C (Fig. 3).

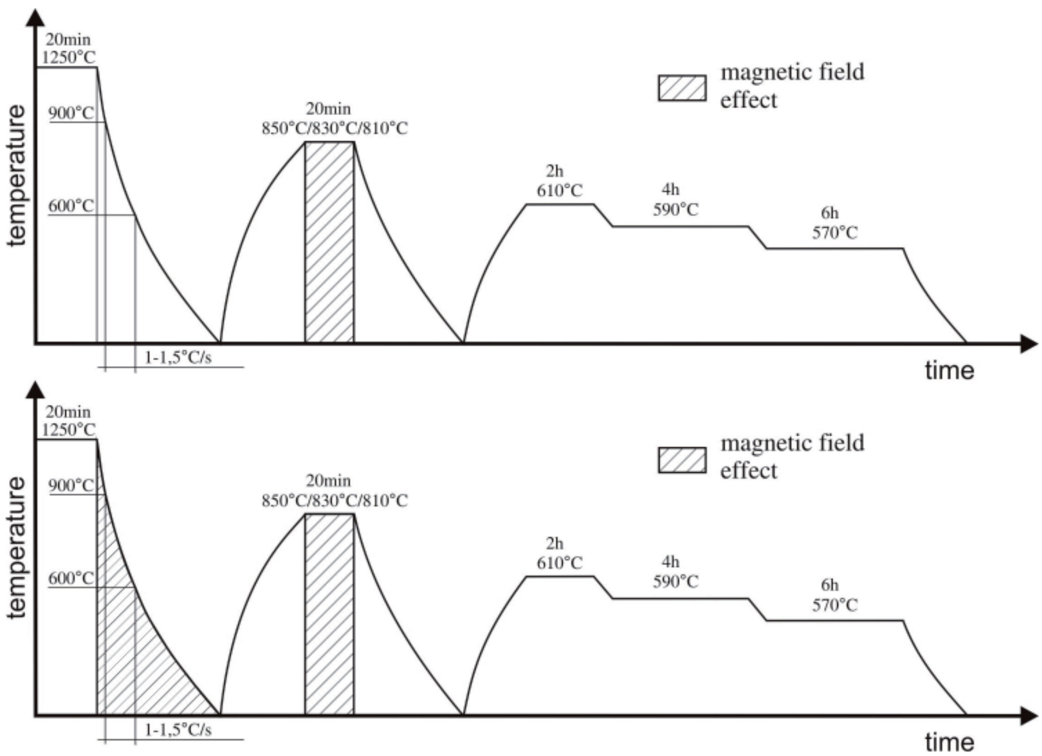

Fig. 3. Scheme of thermo-magnetic treatment of Alnico 8 alloy

The temperature of the isothermal annealing in a magnetic field should be lower than the temperature of the magnetic transition, which for this alloy is 860 °C. The investigations were carried out for the following six variants of the heat treatment:

1. 850 °C for 20 min with cooling after homogenisation without magnetic field,
2. 850 °C for 20 min with cooling after homogenisation in magnetic field,
3. 830 °C for 20 min with cooling after homogenisation without magnetic field,
4. 830 °C for 20 min with cooling after homogenisation in magnetic field,

5. 810 °C for 20 min with cooling after homogenisation without magnetic field,
6. 810 °C for 20 min with cooling after homogenisation in magnetic field.

The structure of Alnico alloys was examined under a Jeol JEM 3010 transmission electron microscope at a maximum acceleration voltage of 300 kV.
The magnetic properties were measured at room temperature using a vibrating sample LakeShore 7410 magnetometer.

3. Results and discussion

The TEM examinations of Alnico alloys undergoing a spinodal decomposition after full heat treatment cycle have revealed the microstructure resembling a chessboard of 30-35 nm side dimensions (Fig. 4). The particles, which are the product of spinodal decomposition, have different magnetic properties – dark fields in the chessboard denote strongly ferromagnetic behaviour, while light fields are weakly ferromagnetic [4]. The electron diffraction pattern taken from the selected area of a specimen shows strong basic reflections corresponding to an RPC phase and weak superstructural reflections. Detailed data from TEM examinations carried out on sintered Alnico 8 magnets are available in [4].

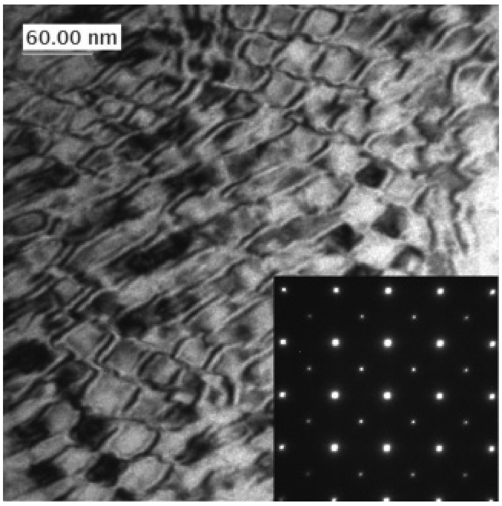

Fig. 4. TEM microstructure of Alnico 8 alloy. The inset shows beside the basic reflections (strong) also reflections from superstructure (weak)

Alnico magnets are characterised by high degree of magnetisation and high remanence but, compared to other types of magnets, their coercivity is relatively low. Considering this fact, the aim of the present study was to investigate possible effect of heat treatment on the maximum increase of coercivity.
Table 1 and Figure 5 show average values of the coercivity obtained for magnets subjected to thermo-magnetic treatment according to the schemes shown in Figure 3. For all the magnets, the remanence varied by approximately 0.8 T. The best results of the coercivity improvement were obtained in samples annealed for 20 minutes at a temperature of 810 °C. In these magnets, the mean value of coercivity remained stable irrespective of whether the magnetic field was applied or not during the homogenisation treatment, and it generally amounted to 132 kA/m for magnets cooled after homogenisation without and with the use of magnetic field. Figure 6 shows typical hysteresis loops for Alnico 8 magnets after homogenisation and thermo-magnetic treatment at a temperature of 810 °C. The hysteresis loops were recorded parallel to the direction of the external magnetic field during the thermo-magnetic treatment. The material after homogenisation has very low properties which only the annealing treatment in magnetic field can improve. For this magnet after a complete treatment cycle, the measured parameters assume the following values: coercivity H_c=133 kA/m, remanence B_r=0.8 T, and maximum magnetic energy $(BH)_{max}$=34 kJ/m^3.

TABLE 1

The results of coercivity measurements for different variants of the thermo-magnetic treatment of Alnico 8 alloy

Temperature of isothermal annealing [°C]	Coercivity ${}_BH_c$ of samples cooled after homogenisation without magnetic field [kA/m]	Temperature of isothermal annealing [°C]	Coercivity ${}_BH_c$ of samples cooled after homogenisation in magnetic field [kA/m]
810	133	810	131
810	132	810	128
810	131	810	135
810	131	810	133
830	116	830	129
830	121	830	126
830	121	830	122
830	121	830	132
850	118	850	124
850	113	850	119
850	106	850	121
850	116	850	121
Temperature of isothermal annealing [°C]	Mean coercivity ${}_BH_c$ of samples cooled after homogenisation without magnetic field [kA/m]	Temperature of isotherma annealing [°C]	Mean coercivity ${}_BH_c$ of samples cooled after homogenisation in magnetic field [kA/m]
810	132	810	132
830	120	830	127
850	113	850	121

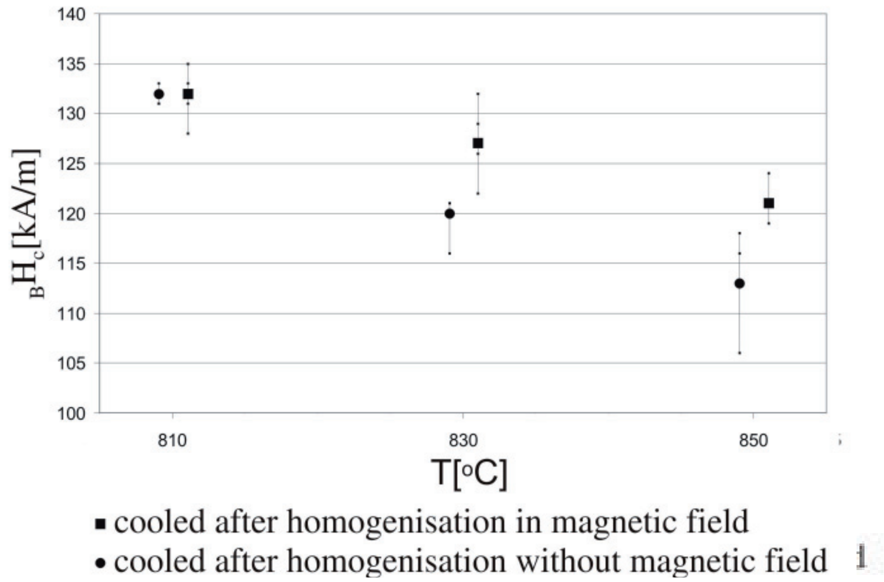

Fig. 5. Effect of thermo-magnetic treatment on the coercivity of Alnico 8 alloy

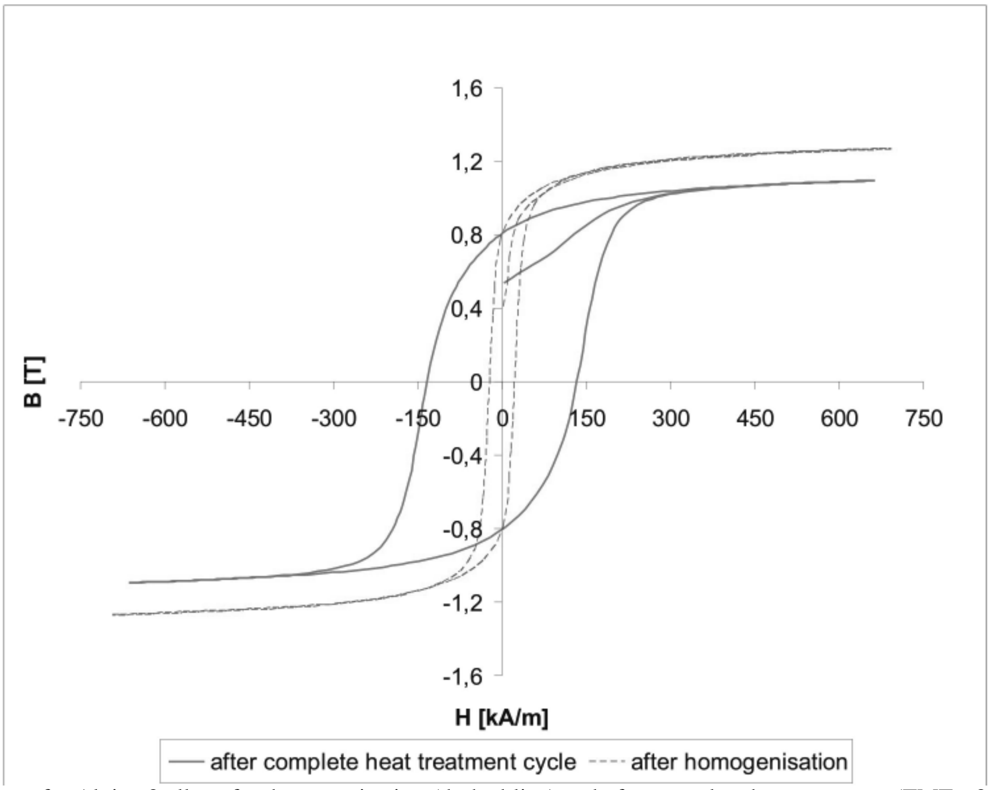

Fig. 6. Hysteresis loops for Alnico 8 alloy after homogenization (dashed line) and after complete heat treatment (TMT - 810 ?C) (continuous line)

At higher temperatures of the isothermal annealing, a drop of coercivity was observed along with the effect of magnetic field during cooling after homogenisation. The average value of coercivity in magnets heat treated at 830 °C amounted to 120 kA/m and 127 kA/m for cooling after homogenisation without and with the use of magnetic field, respectively. After heat treatment carried out at 850 °C, the coercivity was still lower and amounted to 113 kA/m and 121 kA/m for magnets cooled after homogenisation without and with the use of magnetic field, respectively. Hence it follows that when a high-temperature heat treatment is carried out, the use of magnetic field during post-homogenisation cooling is additionally affecting the value of coercivity. Higher values of the coercivity were obtained in alloys cooled after homogenisation in magnetic field. A high scatter in the results of the measurements was also observed. The temperature of thermo-magnetic treatment should be high enough to provide the required rate of diffusion in magnetic field, but even so it should never enter the region of γ_1 phase stability. The temperatures of 830 and 850 °C proved to be too high and caused precipitation of certain amount of the γ_1 phase. It should, however, be remembered that, to some extent, the alloy microstructure is formed during the process of cooling from the temperature of homogenisation, and therefore, in this particular case, the application of magnetic field can improve the properties after a complete heat treatment cycle but certainly not to a degree that would give the same values of the parameters as those that are obtained under optimum heat treatment conditions at 810 °C.

4. Summary

The results of the studies on the effect of homogenisation process and thermo-magnetic treatment parameters show the significant role that the temperature of isothermal annealing plays in obtaining the required coercivity values in Alnico 8 alloy. The highest coercivity was obtained in samples annealed at a temperature of 810 °C. At this temperature, the process of magnetic hardening has proved to be the most effective. The obtained value of coercivity amounting to 132 kA/m is consistent with the values recommended by MMPA No. 0100-00 *Standard specifications for permanent magnet materials*. At an optimum temperature of thermo-magnetic treatment (810 °C), the use of magnetic field during homogenisation is not of great importance. It does improve the properties during homogenisation, when the thermo-magnetic treatment has been carried out at temperatures too high, but even then it shall never allow us to produce magnets which in respect of their service parameters could successfully compete with those treated under optimum conditions.

Acknowledgements

The studies were done under a Research and Development Project No. R 15 012 02, financed from the state budget of the Ministry of Science and Higher Education.

Special thanks are extended by the authors of the study to Magnesy Baildon Sp. z o.o. for making available the samples for examinations and the equipment for heat treatment.

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Received: 10 January 2010.