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Effect of High Pressure on the Al–Mn Binary Alloy System between 40 and 100 at. % Mn

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The effect of pressure on the aluminum–manganese binary system was investigated by preparing various Al–Mn alloys in a cubic multi-anvil pressure apparatus. Pressures of 40 000 and 60 000 atm were used in preparing the alloys from powdered Al and Mn mixes. Indirect heating was used to melt the specimens and to apply a variety of heat treatments (including a magnetic anneal) to the alloys while they were under pressure. The x-ray results indicate that pressures of 40 000 atm have little effect on the Al–Mn system, since the phases prepared under pressure were the same as those found at 1 atm by other investigators of this system. A new identification of the phases present in the η,η' region was made. One phase is body-centered cubic ($a_0=8.9$ Å), the other is face-centered cubic ($a_0=12.5$ Å). It was not possible to ascertain to which phase, η or η', the above a_0 values belonged. The results obtained at a pressure of 60 000 atm differed from those found at 40 000 atm as follows: (a) when quenching alloys in the 50–60 at. % Mn region from 1550°C at 60 000 atm, the magnetic τ phase is formed in preference to the ϵ phase which is formed at 40 000 atm, and (b) a new phase, the structure of which is being determined, was formed from the η,η' phases. Several specimen magnets were made by the high-pressure technique; their magnetic properties were comparable to as-cast AlMn magnets before they were given optimizing treatments.

INTRODUCTION

IN 1958 Kono[1] reported that the compound AlMn was strongly magnetic. Since then there have been a number of investigations made on this compound and the Al–Mn system.[2–4] The interest in this compound is due in part to its tetragonal structure with its high uniaxial magnetic anisotropy, a property that makes it a good permanent magnet material. The purpose of our high-pressure investigation was to determine if extremely high pressures and temperatures could create new Al–Mn phases, preferably magnetic ones, or increase the uniaxial magnetic anisotropy of the AlMn compound.

EXPERIMENTAL PROCEDURE

Pressures of 40 000 and 60 000 atm were generated in a cubic, multi-anvil device,[5] which consists of six anvils, surrounding a cubic block of pyrophyllite, a solid pressure-transmitting medium. Powdered mixes of the desired alloy composition were compressed at 15 000 psi into cylindrical specimens 3.2 mm in diameter by 7.4 mm long. As shown in Fig. 1, the specimen is placed inside the pyrophyllite cube by enclosing it in two cup-shaped pieces of pyrophyllite and placing both pieces inside a tantalum heater tube. Two thermocouples inserted in the cube make contact with the heater. The temperatures reported in this paper are heater temperatures and not specimen temperatures which are 50° to 150°C lower at 800° and 1600°C, respectively. The thermocouple wires are brought out from the center of the cube on adjacent cube faces and make contact directly to the anvil faces. Lead wires attached to the sides of the anvils connect the thermocouples to temperature recorders. Current from a high-amperage–low-voltage power supply passes through the heater and two opposing anvils.

In the initial experiments at 40 000 atm, specimens between 40 and 100 at. % Mn,[6] (at 10% intervals) were given two different heat treatments, i.e., a quench and a slow cool. For the quench treatment the specimens were heated to 1550°C, held for 5 min and quenched to 80°C simply by turning off the power supply. The massive amounts of metal in the pressure apparatus act as a heat sink and quench the specimen at a rate of 100°C/sec. For the slow cool, after holding at 1550°C for 5 min, the specimens were cooled down to 700°C at a rate of 50°C/min and then quenched.

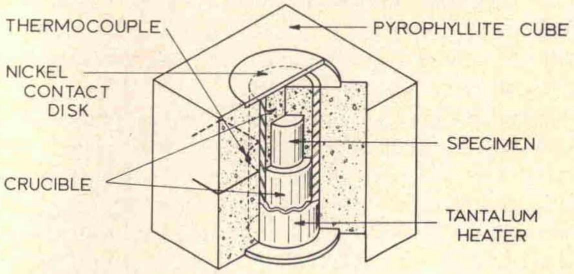

FIG. 1. Schematic view showing the internal construction of the pyrophyllite pressure-transmitting cube.

A second series of experiments consisted of subjecting alloy mixes prepared at intervals of 2% between 48%–56% Mn to the following cyclic heat treatments. The specimens were heated to 1550°C, held there for 5 min, quenched to 80°C, held for 1 min, and then brought back up to 1200°, 1100°, 1000°, or 900°C for 5 min and quenched to 80°C (an annealing treatment). The specimens were then ground into a powder so that x-ray patterns and magnetic moment measurements could be obtained.

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[3] A. J. J. Koch, P. Hokkeling, M. G. v.d. Steeg, and K. J. deVos, J. Appl. Phys. **31**, 75S (1960).

[4] M. A. Bohlmann, ASD-TDR-63-422, Wright–Patterson Air Force Base (May 1963).

[5] A. A. Giardini and E. C. Lloyd, *High Pressure Measurements* (Butterworths, Inc., Washington, D. C., 1963), p. 221.

[6] Compositions are expressed in atomic percent units.

RESULTS

The Al–Mn phase diagram determined by Koester and Wachtel[2] for 1 atm was used to interpret the x-ray diffraction patterns. A pressure of 40 000 atm has little effect on the Al–Mn system since the phases formed under pressure are the same as those reported to exist at 1 atm by other investigators.[2–4] We were unable to find the δ Mn reported by Koester and Wachtel, but this is not an effect of pressure since several 1-atm investigators[3,4] have not found it. The δ Mn phase should have been formed by quenching alloys of the β Mn composition; instead, the only phase present in the quenched alloys between 60% and 100% Mn was the β Mn phase. In the slow cool and anneal treatments, the β Mn phase occurred for compositions containing as little as 56% Mn. Magnetic moment values for these β Mn phase alloys were found to be less than 0.26 emu/g.

For compositions containing 50% to 60% Mn, the ϵ phase was consistently formed by quenching the alloys from above 1200°C, while the slow-cool treatment produced the magnetic τ phase mixed with the β Mn and η, η' phases which are the decomposition products of the ϵ phase. The anneal treatment at 1100°C produced both the τ and ϵ phases in approximately equal amounts. Magnetic moment values obtained for the quenched and slow-cooled alloys were 26.1 and 42.6 emu/g, respectively. These values are lower than the 96.0 erg/Oe-g value obtained by Koch.[3] This is not surprising since the specimens were not pure τ phase material but contained other phases due to the nonuniform temperature conditions in the high-pressure cell. The β Mn and η, η' phases were found in alloys given an anneal below 1100°C.

Below 50% Mn, η and η' were the only phases found. Two new structures were determined for these phases but it was not possible to ascertain to which phase η or η' the structures belonged because the two phases were always found together and one could not be prepared without the other. The lattice constant for one of the phases was determined from a single crystal selected from a η, η' melt and it was found to be body-centered cubic ($a_0 = 8.95$ Å). The remaining phase in the combined η, η' powder pattern was then identified as face-centered cubic ($a_0 = 12.5$ Å). However, Koch[3] has reported the structure of the η phase to be hexagonal and that of the η' phase as unknown. All of the heat treatments formed this mixture of the η and η' phases which had magnetic moments ranging between 0.33 and 0.44 emu/g.

The 40 000-atm quench experiments were repeated at a pressure of 60 000 atm which does have an effect on the Al–Mn system. First, the alloys quenched from above 1550°C in the 50%–60% Mn region contained more of the magnetic τ phase than those prepared at the lower pressure. This was indicated in both the x-ray data and the magnetic moment data where the moment was increased from 26.2 to 65 emu/g. Second, the η, η' phase region is transformed into a new phase region, the phases of which have not yet been determined.

Several cylindrical AlMn (52% Mn) magnets, 3.2×7.4 mm, were prepared from the elemental powders in four different ways under pressure. Magnets A, B, and C were prepared at 40 000 atm using the 1100°C anneal treatment, A and B for 5 min, C for 2 h. Magnet B also had a dc magnetic field of 80 Oe applied to it while under pressure by winding a field coil on a pyrophyllite form and placing it around the heater. Magnet D was prepared with a quench treatment at 60 000 atm and without an applied field. The magnetic properties of the magnets are tabulated in Table I. Although these values are lower than those obtained for the best AlMn magnets,[4] they compare favorably with untreated as-cast magnets. However, only a few of many possible treatments usually used to improve the magnetic properties of a material were attempted.

TABLE I. Magnetic properties of several cylindrical AlMn (52% Mn) magnets.

Magnet	$4\pi I$	B_r	H_{ci}	$(B_dH_d)_{\max}$
A	3000 G	880 G	740 Oe	0.10 (10^6) GOe
B	3700 G	1600 G	850 Oe	0.31 (10^6) GOe
C	4800 G	1600 G	750 Oe	0.29 (10^6) GOe
D	4400 G	1600 G	830 Oe	0.36 (10^6) GOe

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