WATER ATOMIZED Fe-Co-BASED POWDERS FOR Alnico MAGNETS

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KEY WORDS: permanent magnets, alnico magnets, magnet manufacturing, metal powders, Fe-Co powder, powder preparation, water atomization, powder compaction, powder sintering, magnetic properties

ABSTRACT: Sintered Alnico magnets remain commercially of interest and therefore a subject of R&D works in spite of increased atractivity of new rare-earth super magnets. Sintered AlNiCo anisotropic permanent magnets are produced from a mixture of powders. The master alloy powder is produced conventionally by grinding and milling of a cast alloy. Our aim was to replace this procedure with technologically, economically and ecologi cally a more effective powder preparation method. We therefore investigated the applicability of water atomization for the preparation of Fe-Co-based metal powders. The appropriate chemical compositions and process parameters were determined on the basis of the significant features of water atomization. The morphological properties of the prepared powders were determined as functions of the main influent parameters of water atomization. Water atomized powders were then mixed with an appropriate amount of different additions and compacted into green samples. Samples were vacuum sintered and thermomagnetically treated under different conditions on the laboratory and industrial scale. The resulting magnetic properties of the prepared materials are equal in comparison with commercially available samples and better than those of conventionally prepared materials.

Vodno atomizirani Fe-Co prahovi primerni za izdelavo Alnico magnetov

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KLJUČNE BESEDE: magneti trajni, magneti alnico, izdelava magnetov, prahovi kovinski, prah Fe-Co, priprava prahov, atomizacija vodna, stiskanje prahov, sintranje prahov, lastnosti magnetne

POVZETEK: Kljub temu, da danes večino raziskovalcev s področja magnetnih materialov privlačijo predvsem še nepojasnjeni pojavi in nadaljne izboljšanje lastnosti redkozemeljskih supermagnetov, ostajajo tržno in zato tudi raziskovalno še vedno zanimivi, že dolgo uveljavljeni trdomagnetni materiali, kot so feriti in tudi Alnico magneti. Drobljenje, mletje in sejanje ulite predzlitine je ustaljen in precej zastarel postopek priprave kovinskih prahov za izdelavo AlNiCo anizotropnih trajnih magnetov. Ta postopek smo zato želeli zamenjati s tehnološko, ekonomsko in ekološko učinkovitejšim postopkom. Zato smo raziskovali možnosti uporabe postopka vodne atomizacije za izdelavo prahov na osnovi kobalta in železa primernih za izdelavo AlNiCo anizotropnih trajnih magnetov. Praktično delo nam je omogočila predvsem nova laboratorijsko pilotna naprava za vodno atomizacijo z induktivnim talilnim sistemom. Glede na tehnološke značilnosti postopka vodne atomizacije smo izbrali primerno kemično sestavo vložka in določili osnovne tehnološke parametre atomizacije. Izdelanim prahovom smo določili osnovne morfološke lastnosti v odvisnosti od parametrov atomizacije. Lastnosti izdelanega prahu so bile v okviru pričakovanj in tehnoloških možnosti postopka vodne atomizacije. Izdelanim vodno atomiziranim prahovom smo primešali potrebne dodatke za korekcijo sestave in izboljšanje stisljivosti in jih nato stiskali v preizkusne valjčke. Stisnjene surovce smo nato sistematično sintrali in toplotno obdelali v magnetnem polju pri različnih pogojih tako v laboratoriju, kot tudi na industrijskih napravah. Dosežene magnetne lastnosti obeh preiskovanih materialov (tip 400K in 1500) so bile podobne komercialnim vzorcem te vrste.

INTRODUCTION

Standard procedure of Alnico (Al-Ni-Co-Fe based alloys) permanent magnets production in ISKRA Magnets factory, Ljubljana, Slovenia is based on melting and casting of Fe-Co master alloy with additions of other alloying elements, such as Ni, Al, Cu, Si, Nb and Ti. The next production step is powder fabrication via grinding, milling, sieving (see **Fig. 1**) and mixing with a proper amount of binder and lubricant for automatic die compaction. Compacted green parts are then sintered in a protective atmosphere or vacuum, as well as thermomagnetically and mechanically treated.

Nowadays it is possible to produce metal powders with more effective powder preparation methods. The most important are the physical methods of powder preparation and among them fluid atomization (inert-gas and water atomization) is the most wide spread. The atomization or powder production directly from melt has many

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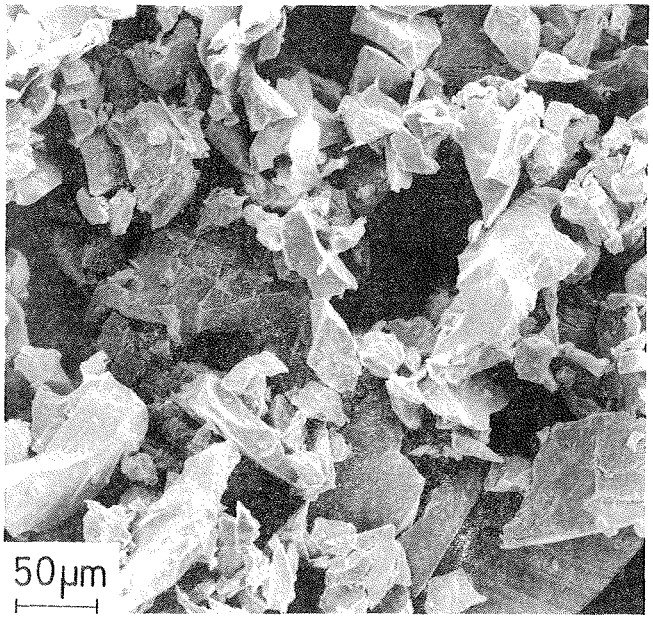

Fig. 1: SEM micrograph of the powder prepared by conventional powder fabrication method, Fraction: ≤ 63 µm

advantages. In particular, casting, grinding and milling of master alloy is not necessary. For a powder particle size in the 10 to 100 µm range, the cooling rate of disintegrated particles during water atomization is estimated on approximately 10^2 to 10^6 K/s. This means that we can expect better chemical homogeneity and microstructure refinement because of rapid solidification. Another advantage of water atomization is simple control of mean powder particle size by water pressure and relatively narrow particle size distribution of produced powder. On the other hand we can expect some difficulties in preparing metal powder with the selected final chemical composition because of oxidation of presented reactive elements such as Al, Ti and Nb during water atomization, available for our experiments. Also, we did not find out references[1,2] yet, that it is possible to prepare powder by water atomization, appropriate for the production of sintered Alnico permanent magnets.

EXPERIMENTS AND RESULTS

For our R&D work, two conventional hard magnetic materials were selected. The first one has trade-mark SIMAG 1500 (equivalent to DIN Alnico 39/12) with the nominal remanence Br = 0.85 T and coercivity H_{cB} = 118 kA/m and the other one is SIMAG 400K (DIN Alnico 28/6) with Br = 0.9 T and H_{cB} = 55 kA/m. Water atomized powders with various chemical compositions were prepared by new pilot water atomizer (D5/2 Davy McKee, England, recently installed at the Institute of Metals and Technologies in Ljubljana, Slovenia) at different process parameters of atomization to optimize process and powder properties. All prepared metal powders were examined by light microscope and SEM (Scanning Electron Microscopy). Basic physical and morphological properties (particle shape, mean particle size, particle size distribution, apparent density and flowability)[3] of powders were also determined. In **tables 1** and **2** are collected the most important controlled parameters of water atomization.

With the assistance of classical chemical analysis and X-ray fluorescence melts and final chemical compositions of atomized powders were controlled and corrected, respectively. Oxygen content of prepared powders was between 0.1 and 0.15 wt.%. Another very important parameter in chemical composition of AlNiCo magnets is carbon content which does not exceed 0.05 wt.%. All prepared powders have irregular particle

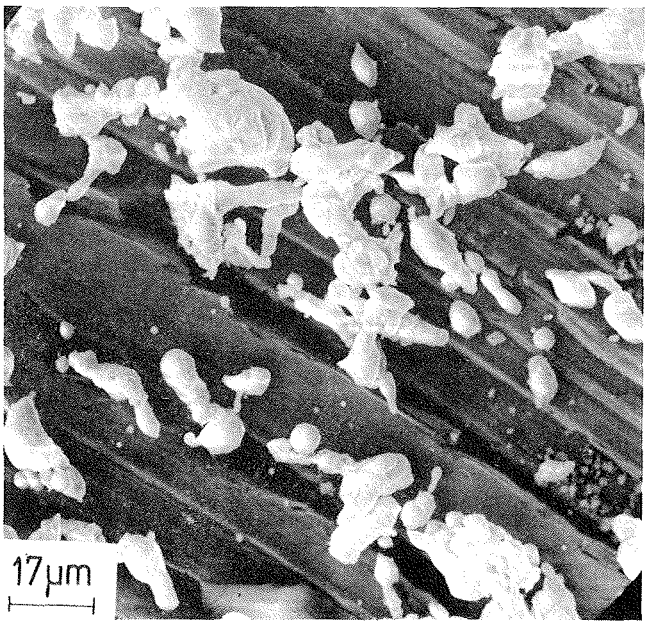

Fig. 2: *SEM micrograph of Alnico water atomized AlNiCo powder. Fraction: ≤ 63µm*

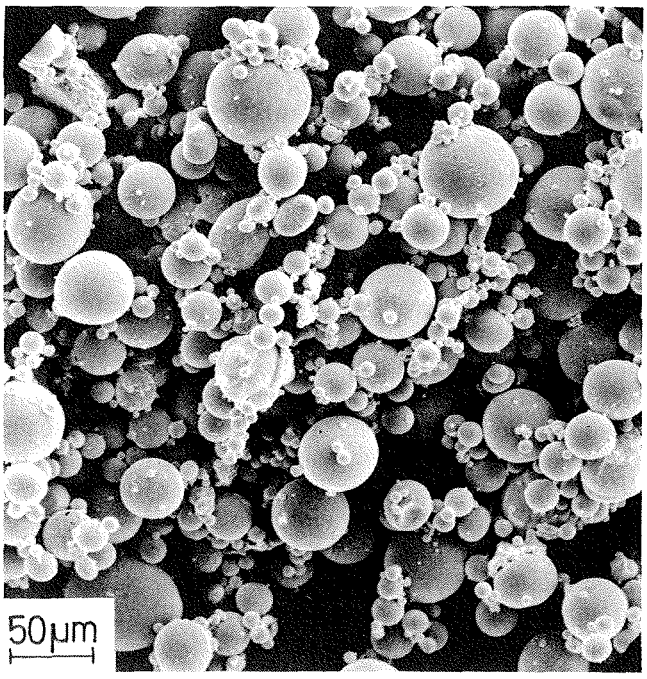

Fig. 3: *SEM micrograph of gas atomized AlNiCo 1500 Fraction: ≤ 63 µm*

shapes (see **Fig. 2**) which is characteristic of water atomization[2,4]. This means that we can expect better compressibility but lower flowability of water atomized

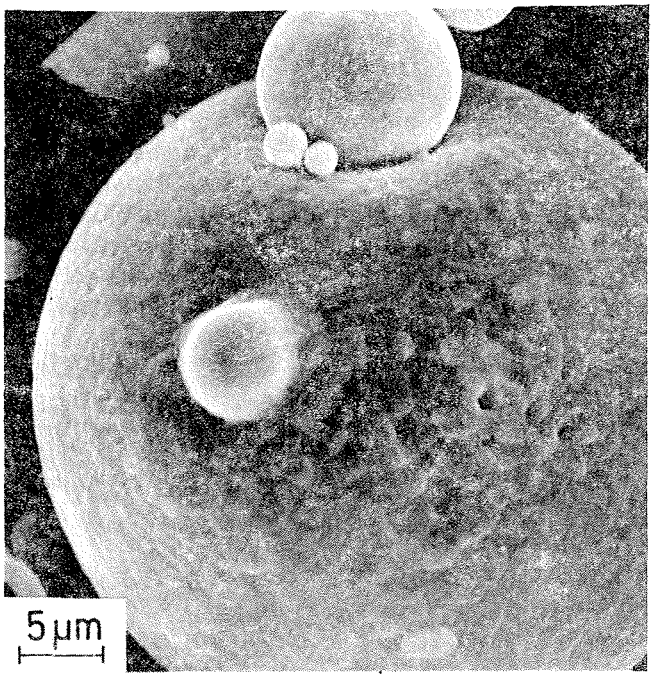

Fig. 4: *SEM micrograph of particle of gas atomized AlNiCo 1500 powder.*

powders compared with gas atomized powders (see **figures 3 and 4**).

Mean particle size of prepared powders varies between 45 and 100 µm, depending on selected chemical composition, superheating and water pressure. For consolidation only powder fraction ≤ 120 µm was used. The optimal chemical composition was selected on experimental basis, so that the combination with other additions (Ti-hydrid powder was replaced with cheaper CoTiAl or NiTiAl commercially available powders) prevents oxidation of the most reactive elements during water atomization. The compressibility of powders was improved with the reduction of iron content in water atomized powders and adding of soft pure Hoeganaes iron to powder mixtures.

The systematical experimental cold pressing (600 to 750 MPa with 0.5 wt.% wax) and sintering (in vacuum 10^{-2} mbar, 1350 to 1370°C and 1 to 3 hours) of prepared water atomized powders in mixtures with other additions were carried out on laboratory and industrial scale. Green density of samples was between 5.7 and 6 g/cm^3 and sintering density was from 7.05 to 7.30 g/cm^3 depending on pressing and sintering conditions, respectively. The main magnetic properties were determined after thermomagnetical and mechanical treatment of samples.

Obtained average magnetic properties of sintered Alnico magnets prepared from water atomized powders are presented in **table 3**.

Table 1: Experimental process parameters for preparation of Alnico powders types 400K and 1500 (Davy Mc KeeD5/2 water atomizer - IMT Ljubljana).

Process parameters		Remarks
Superheating of melt (°C)	1550 to 1650°C	measured by optical pyrometer
Temperature of tundish (°C)	1250 (± 20°C)	thermocouple Pt-PtRh13
Tundish nozzle diameter (mm)	4.5	fused quartz
Water jets diameter (mm)	main 1.20 x 1.05 side 1.10 x 0.85	type 1503 type 1502
Jet apex angle	main 25° side 20°	original manifold
Water pressure (bars)	180 to 230	
Flow rate of protective gas	0.8 m^3/h N_2 in atomiz. chamber 0.3m^3/h Ar over melt during melting	flow-meter

Table 2: Parameters of water atomization for Alnico powders (Davy McKee D5/2 water atomizer-IMT Ljubljana).

Material	Water pressure (bars)	Water flow rate (l/min)	Metal flow rate (kg/min)	Flow ratio (water/metal)	d_{50} (µm)
SIMAG 400K	180	56	3.54	15.82	50
SIMAG 1500	200	55	6.65	8.30	60

Table 3: Magnetic properties of sintered Alnico magnets prepared from water atomized powders.

Material	Magnetic properties			Contents	
	B_r (T)	H_{Cr} (kA/m)	$(BH)_{max}$ (kJ/m^3)	wt.% O_2	wt.% C
SIMAG 400K	1.10	60.0	28.5	0.10	0.050
SIMAG 1500	0.90	124.0	42.0	0.15	0.035

It is necessary to point out that besides the above mentioned advantages of water atomization, water atomized powders have different (**see figure 5**) and better sinterability, respectively. This results in finer microstructures with narrower grain size and pore distribution (**see figures 6 and 7**). The final result are more uniform magnetic properties and improved dimensional stability of sintered Alnico products.

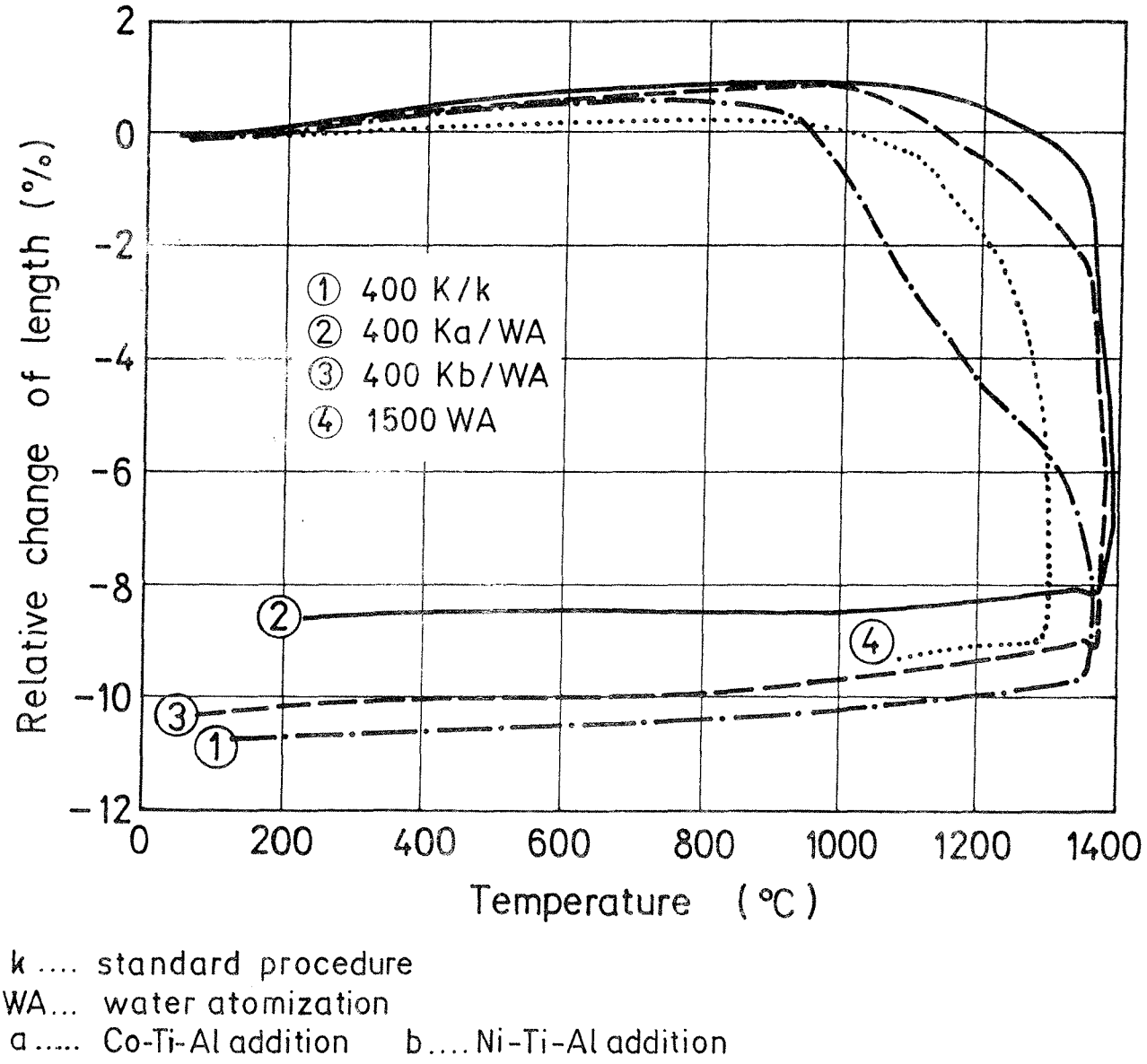

Fig. 5: *Comparison of dilatometric curves for conventionally prepared Alnico powder type 400K and powders prepared by water atomization.*

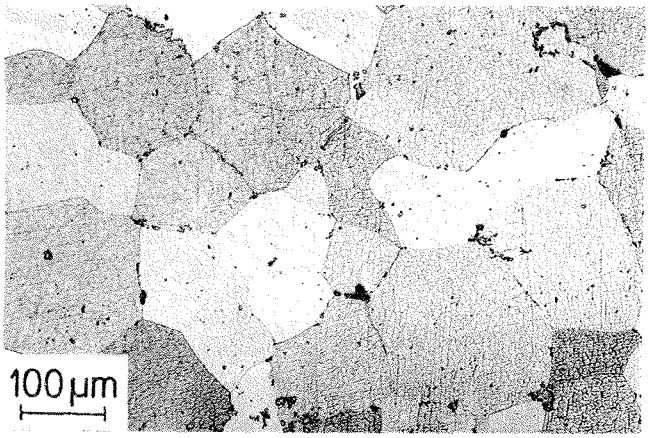

Fig. 6: *Optical micrograph of the typical microstructure of sintered Alnico type 400K magnet prepared by standard procedure.*

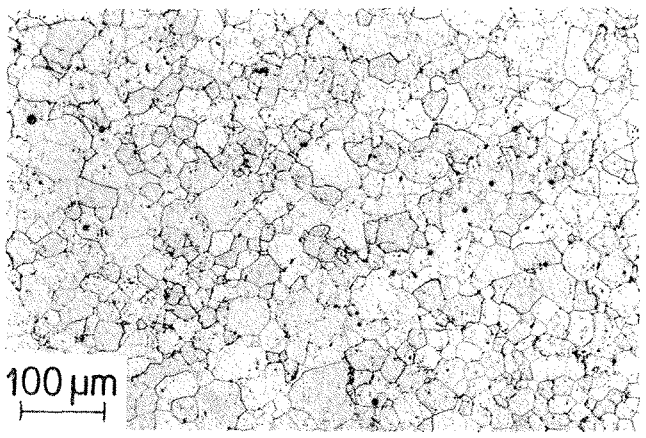

Fig. 7: *Optical micrograph of the typical microstructure of sintered Alnico type 400 K magnet prepared from the water atomized powder.*

CONCLUSIONS

We investigated the applicability of water atomization for the preparation of Fe-Co-based metal powders suitable for the production of Alnico permanent magnets. On the basis of the significant features of water atomization the appropriate chemical compositions and process parameters were selected. The basic physical and chemical properties of prepared water atomized powders were determined. Prepared powders were then mixed with other commercially available powders and additions, pressed and sintered at different conditions, thermomagnetically treated and ground. The measured magnetic properties of the prepared sintered AlNiCo magnets are equal in comparison with commercially available samples and better than those of conventionally prepared materials.

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