Poster | Raw materials

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## [P2] Raw Materials & Recycling

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# [P2-22] Melting and solidification behavior of oxidized Nd-Fe-B powder upon plasma spheroidizing

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Currently, the development of 3D printing technology, in particular, the selective laser melting of NdFeB permanent magnet blanks is an urgent scientific and technical problem. As materials for the realization of such a technology, spheroidized powders prepared by melt sputtering or via spheroidizing an alloy of a given composition, which was preliminary prepared by strip-casting and subsequently milled, are used. After the end of life of various devices, a great amount of end-of-life permanent magnets are collected, which have no further practical application and are industrial wastes. At the same time, the use of the secondary materials in fabricating the Nd-Fe-B permanent magnets (so-called recycling) will allow one to substantially decrease the first cost of the magnets. As the initial powder, we used crushed scrap (prepared by crushing followed by milling) of out-of-life sintered NdFeB permanent magnets having the following composition (wt %): 1.18B, 25.5Nd, 6.5Pr, 1.5Dy, 0.7Tb, 0.7Co, 0.65Al, and Fe balance; the oxygen content is to 1.85 wt%. Before spheroidizing, the powder was sieved to separate powder 30-63 μm in particle size. The powder was spheroidized using a plasma generator designed in the Baikov Institute of Metallurgy and Materials Science, Russian Academy of Sciences. Powder particles and present oxide inclusions are melted under the action of plasma jet and form two immiscible liquids, namely, metallic and oxide (slag). These liquids actively react with each other; in this case light REMs transfer from the metal melt into the slag (Fig. 1d,f). The active separation into two liquids occurs also upon solidification of melt drops. After leaving the plasma, melt drops enter in the medium that ensures the very high cooling rate (more than $10^5$ K/s). In accordance with the particle size, namely, 8-12, ~25, and ~60 μm (Fig. 1a), the different behavior of the melt upon solidification can be observed. In the case of fine particles 8-12 μm in size (their cooling rate is highest), the separation has no time to occur; the particles contain both light and heavy REMs, and the $(Nd,Pr,Dy,Tb)_2Fe_{14}B$ phase can be formed without primary solidification of iron. In the case of a particle ~25 μm in size (intermediate cooling rate), the separation begins to occur, and the slag is enriched in light REMs. However, slag has no time to enter to the particle surface and is present inside the particle in the form characteristic areas. In the case of large particle, ~60 μm in size (the lowest cooling rate among the considered particles), the slag had time to enter to the particle surface upon solidification. In the case of ~25 and ~60 μm particles, for which the transition of light REMs to the slag is observed, the total content of REMs in the metallic melt decreases, whereas the iron content increases; this ensures the primary solidification of iron in the form of very fine particles, which is not suppressed even by very deep supercooling of the melt realized in the course of spheroidizing (homogeneous nucleation). In this case, the iron content in the final spheroidized powder is 50-52 wt %, whereas the slag is almost free of iron (Fig. 1b). After primary solidification of iron, the 2-14-1 phase is formed in these particles. In the case of initial powder fraction of 20-30 μm, 2-14-1 phase content in spheroidized powder is 22-23%, and the oxide content is 15 wt % (the oxygen content is to 6 wt %). In this case, oxides are observed in the form of a slag at the particle surface. The obvious enrichment of the melt with heavy REMs at the expense of leaving Nd and Pr of the metallic melt and their transfer to the slag is observed (Fig. 1c,e). Thus, formed spherical particles are the mixture of very fine iron particles and 2-14-1 phase enriched in the heavy REMs. In the course spheroidizing, the active evaporation of elements from the metallic melt occurs, which leads to changing the melt chemical composition. The study was performed in terms of state assignments no. 075-00320-2400.

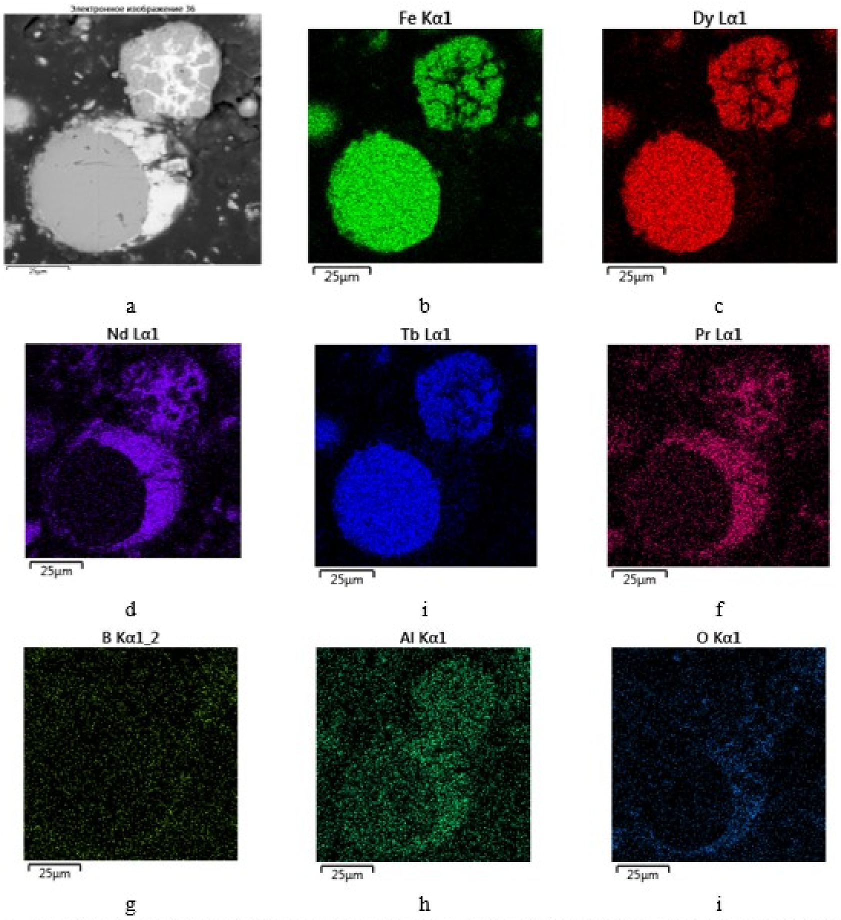

Fig. 1. (a) SEM (reflected electron mode, EDS) image of the typical microstructure of spheroidized particles Nd-Fe-B and (b-i) element distribution maps.