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Peculiarities of selective laser melting process for permalloy powder

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ABSTRACT

The new soft magnetic material based on the permalloy powder was successfully used for a three dimensional sample fabrication via selective laser melting process. The microstructures of the Fe-80%Ni-alloy samples as a function of the laser influence parameters were studied by optical and scanning electron microscopes equipped with energy-dispersive microanalysis, X-ray diffraction and micro hardness measurements. The effects of external magnetic fields (EMF) on the growing structures during the laser melting were estimated. The reduction in coherent-scattering regions of magnetic phases under SLM with EMF is accompanied by the coercivity growth.

1. Introduction

Magnetic iron–nickel alloys known as permalloys are of great interest due to their remarkable magnetic properties [1]. The permalloys are notable for their low coercivity, high permeability and high magneto-conductivity in weak magnetic fields that play a key role in sensors, transformers, inductive device, electric motor, etc. [2–4]. A giant magnetoresistance (GMR) in low fields of about 10 Oe was observed at room temperature in as-prepared laser-deposited permalloy/nonmagnetic multilayers [5]. A significant columnar growth for the GMR could be controlled under a precise doping of laser energy only.

The conventional powder metallurgy (PM) methods of casting and machining are hardly suitable for realization of mass production of miniature magnetic devices and those of complex shape, due to the required secondary operations such as micro-machining, grinding and drilling, etc. Moreover, traditional PM techniques such as high-temperature sintering and thermal spraying (temperature cooling rate, TCR$\sim 10^2$–10^4 grad/s) may lead to the decrease of magnetic properties due to the excessive grain growth under a low speed thermal heating as compared with the laser treatment (TCR$\sim 10^3-10^5$ grad/s). Lately a number of new methods were proposed for fabrication of magnetic devices, including metal injection moulding [6,7], spark plasma and arc discharge sintering [8,9] and mechanical alloying with the following compaction [1,10–13]. However, the soft magnetic permalloy cores are produced by these methods in the limited form of toroidal coils and stacked ribbons or thin sheets [11]. Therefore, efforts must be devoted to prepare the complex samples by original powder process.

From the standpoint of the magnetic properties conservation, the selective laser sintering/melting (SLS/M) is a promising technique for fabricating of functional and miniature devices and multilayer graded structures with alternated properties and perspective applications [2,14–18]. The authors did not find any literature data about the laser parameters influence on microstructure under the effect of external magnetic field (EMF). Interpretation of results in the relevant study [2] has a number of problems connected with doubtful nano size of grains after cw laser influence and unobvious crystallite size trend for combined magnetic phase conglomerations. The present study is dedicated to the search of optimal conditions for the direct SLM of the permalloy powder, to finding out whether there is any influence of EMF on the growing structures during solidification and fabricating of samples with nearly full density, and to the microstructure evaluation.

2. Experimental procedures

The powdered permalloy PR-81N3M (Polema Ltd., Tula, RF) was used as the source material and had the following chemical composition: Mo 2.9 wt%, Mn 1 wt%, Ni 80.6 wt%, balance Fe. The powder size distribution was pre-sifted on the Retsch AS 200 (Retsch GmbH, Germany) and studied by means of the ALPAGA 500NANO (OCCHIO) granulomorphometer. The particle size parameters were estimated to be of the following equivalent diameter

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(weight by volume) d_{10}=33.2 μm, d_{50}=74.2 μm, d_{90}=118.2 μm and 70% of particles had equivalent diameter (by number) more than 73.9 μm. While 10% of particles had the shape factor less than 81% and roundness of about 84%. The substrates were round plates made of steel with the 50 mm-diameter and 5 mm-height.

In the beginning, the optimal parameters of the SLM process on air were determined for the PR-81N3M permalloy on the laboratory setup for the layering SLM [14,18]. Prealloyed magnetic powder and absence of specific environment make our study different from [2]. Parameter ranges obtained for fabrication of the 3D parts were the following: laser power, P=20–150 W; scanning speeds, V=5–20 mm/s; hatch distance between the laser passages, Sh=50–150 μm; layer thickness of the delivered powder - H=80–130 μm. To obtain the EMF we used a constant magnet with the magnetic field ~1 T. The laser beam diameter D was 100 μm. We used a combined parameter of the specific laser energy input (SLEI) which was determined as $E=P/(V^*D)$ [J/mm^2] for the LI optimization. The successful resulting 3D cube object obtained is shown in graphical abstract figure.

It was necessary to select the etching agents for the prepared micro sections in order to maximally reveal the basic structural phases. For our Fe-Ni system, aqueous solution of ferrous chloride and nitric acid taken in equal portions has proved to be convenient. After the etching, cross sections of multi-layered melting samples were subjected to metallurgical analysis with the optical microscope (Neophot 30 m, Carl Zeiss) equipped with a digital camera. The 3D samples obtained under the optimized regimes, were analyzed by optical microscopy (Olympus BX51 M, Japan), PMT-3m (OKB SPECTR Ltd., St. Petersburg, Russia), microhardness testing and scan electron microscopy (VEGA 3 LMH, Czech Republic) equipped with the EDS microanalysis, and LEO 1450 scanning electron microscopy (Carl Zeiss Company) equipped with an energy-dispersive x-ray analyzer (INCA Energy 300, Oxford Instruments). The phase composition of the SLM parts was determined by XRD with the use of a DRON-3m (Bourevestnik Inc., St. Petersburg, Russia) diffractometer in Co-K$_\alpha$ radiation.

3. Results and discussion

Initially the optimal regimes for individual SLM passages were determined. The resulting tracks were evaluated by their external appearance and then a stable zone was identified by the method proposed in [14,18]. Then these regimes were used for further experiments under the influence of the external magnetic field and without it by means of the transverse sections preparation.

Fig. 1 presents the results of optical metallography for successful cladded layers on the substrate with the LI regime P=100 W, V=20 mm/s, Sh=80 μm. Laser passage strips in the cross-section direction are clearly visible in Fig. 1. The permalloy remelted microstructure (Fig. 1) has lamellar elements characteristic for multipass laser passages; these elements represent the cross section of the separate rollers of the melted seam. The arrow in Fig. 1 indicates a spherical void formation that testifies to problems with the above mentioned LI regime. The dendrite or cellular structure features are not explicitly visible.

The experimental measured microhardness of the laser melted layers was estimated in the range of 180–215 HV$_{0.1}$ and it was irregular. Microhardness fluctuation is connected with the grain boundaries and local porosity registered in Fig. 1. The large number of grain/particle boundaries causes an increase of electrical resistivity and diminution of the coercivity [19]. A further increase in resistivity may be possible due to incorporating structure porosity. However, the porosity must be closed so that permalloy oxidation would not result in a reduced saturation magnetization. As a whole the microhardness values correspond to those similarly measured for the FeNi alloys [4].

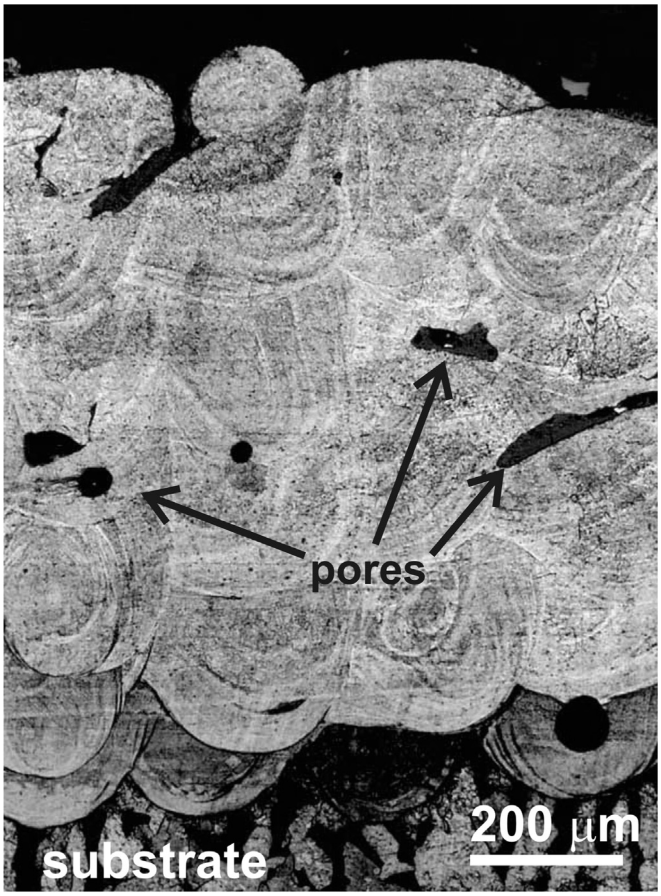

Fig. 1. Optical microscopy image for common view of cross-section with pore's inclusions (×100). Arrows had shown a void's placement.

The X-ray analysis results are presented in Fig. 2a. As it is seen, the most intensive lines belong to taenite-(111) FeNi and awaruite-(111) FeNi$_3$ intermetallic phases. Less intensive (200), (220) lines belong to FeNi$_x$ intermetallides and magnetite Fe$_3$O$_4$ (311) and (551) on the remote angles. A similar intensity line distribution was observed in study [9] after the spark plasma sintering and in [15,17] after the SLM process for the Fe-80%Ni mixed powder. In study [16] five of the similar peaks produced by bulk FeNiMo were indexed to the following lattice planes: (222), (311), (220), (200) and (111) by comparing them with those listed in the International Centre for Diffraction Data powder diffraction file for awaruite NiFe (PDF card 12-0736), which seems to be the closest to FeNi$_3$ phase (PDF card 38-0419) basing on the available X-ray diffraction information.

It was also an object of interest to understand how the nature of intensity peaks changes with the growth of the SLEI – E, and in case of the EMF effects upon the results during the SLM process. So under the laser power of P=60 W and with the increase of scan velocity V (regimes 1–3, Fig. 2a), the SLEI falls, that is confirmed by a decrease of the diffraction peak intensities. With a constant scan velocity V=20 mm/s and increase of the laser power P=60–100 W (regimes 3–4), an insignificant growth of the SLEI (from 30 up to 50 J/mm^2) takes place, and the peak intensities are also quite weak. It correlates well with the SLM regime range and the distribution of the phases in studies [15,17].

And last, the EMF-switching-on intensifies the formation of magnetite Fe$_3$O$_4$, known to have a very strong magnetization. In this case the intensity peaks for basic FeNi and FeNi$_3$ intermetallide phases are also growing (curve 5, Fig. 2a). Thus, we can