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Study of magnetoelastic properties of pure nickel parts produced by metal injection moulding

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A B S T R A C T

The production of smart materials such as those with magnetoelastic properties is of increasing interest for some application as position transducers. The use of the metal injection moulding process permits the production of small parts with complex geometries as well as avoiding common defects produced by other processing techniques. In this study, a feedstock based on pure nickel and thermoplastic binder has been designed and moulded to have specific cylindrical geometry and defect-free green specimens. The parts were sintered changing several processing parameters that have influence on magnetoelastic effects. These effects were studied in terms of the field-dependent elastic modulus, which was estimated by subjecting the specimens to free longitudinal vibration while they remained within different magnetic fields from 0 to 2000 Oe. The optimal sintering parameters turned out to be 1325 °C for the temperature and 12 h for the holding time, whereas the estimated field-dependent elastic modulus variation (8%) was higher in comparison to variations in parts obtained by conventional processing (4%).

1. Introduction

Magnetomechanical materials are those which show a reciprocal coupling between their mechanical and magnetic properties. More specifically, Jiles emphasizes two different types depending on the reversibility of the induced variations [1]: if the altered properties do not recover their initial state after removing the magnetic or mechanical action, the material is a magnetic shape memory alloy; otherwise, the material is called magnetoelastic.

All magnetic materials, and magnetoelastic ones in particular, are currently of topical interest due to their excellent features for many advanced and cutting edge applications [2]. They are used in sonar systems for underwater submarine detection; as transducers in ultrasonic actuators for micro-positioning and vibration control of heavy structures and industrial machinery; as transducers in magnetostrictive position sensors; in the development of inchworm motors and wireless linear micro-motors, etc.

Magnetoelasticity is only significant in magnetically ordered materials such as ferromagnets due to their inherent structure of magnetic domains [3]. Some alloys have been developed in order to obtain giant-magnetoelastic effects (in particular, giant-magnetostriction), such as Terfenol-D and Galfenol, but the role of the classical ferromagnetic materials (iron, nickel and cobalt) is still important since they are still used and they are the major constituents of most soft magnetic alloys [2]. Thus, this work is focused on nickel (since its magnetoelastic behaviour is better than that exhibited by iron and cobalt [3,4]) and on the study of the dependence of its elastic modulus on the applied magnetic field (the so-called ΔE-effect) since it can provide valuable information about the magnetoelastic capacity of materials [4,5]. Being more specific, ΔE-effect will be described in terms of the ratio, as shown in Eq. (1):

$$\frac{\Delta E_{sd}}{E_d} = \frac{E_s - E_d}{E_d} \qquad (1)$$

with E_d and E_s being the demagnetized and saturated Young's modulus, respectively.

Nickel and nickel alloys are commonly produced by conventional manufacturing processes, such as casting, forging and machining [6]. These processes are associated with problems that can affect the final properties of the part, such as the concentration of alloying elements on grain boundaries in the casting process, the grain orientation in the forging process, residual internal stresses, costs and elevated losses of material in the machining process, and in many cases final heat treatments are required to improve the functionality of the part. Nickel based alloys are known as some of the most difficult-to-machine alloys in order to satisfy production and quality requirements [7,8]. Although nickel-based alloys are not exceptionally hard, their outstanding high temperature strength and extreme toughness create difficulties during machining due to their work hardening tendency which results in

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very high cutting forces and significant burr formation during machining [9]. For this reason, mass production of miniaturized parts with complex shapes is greatly limited due to the long production period, low efficiency and high cost. In addition to the surface alteration induced during machining some residual stresses may result in a distortion which adversely affects the mechanical properties, stress-corrosion properties of the machined components [9] and magnetic properties. To solve these problems and in order to obtain fine grains and uniform distribution of microstructure, improved toughness and machinability, removing internal strains, increased wear resistance, improved response to heat treatments, much more precise adjustment and homogeneity of the composition and obtaining of unobtainable alloy by conventional methods some novel manufacturing methods and advanced techniques such as powder metallurgy (PM) are employed [10].

Metal Injection Moulding (MIM) is a powder metallurgical forming technique very suitable for economical mass production. Furthermore, MIM is a near net shaping technique that is particularly advantageous for applications where complex shapes with high dimensional accuracy and high density are required [11,12]. In addition, MIM is used to obtain fine grains and a uniform distribution of microstructure, improve toughness and machinability, remove internal strains, increase wear resistance, improve response to heat treatments, adjust and distribute the composition more precisely and obtain alloys which would be unobtainable by conventional methods.

In the last few years, numerous studies have focused on achieving pieces with magnetic properties through the application of MIM technology in nickel–iron alloys [13–19]. Other more innovative studies deal with materials alloyed with rare earths which enhance their performance, like gadolinium which shows a paramagnetic or ferromagnetic behaviour depending on the temperature [20]. However, studies on magnetic and magnetoelastic properties of pure nickel fabricated by MIM have not been found. For all these reasons, it is very interesting to apply the MIM technology for obtaining magnetic nickel pieces with high mechanical properties. In this paper pure nickel was produced by MIM and the influence of several significant processing parameters on the magnetoelastic performance was reviewed. Factors that influence the magnetoelastic properties such as density and grain size were analysed.

2. Experimental procedure

This research is based on a previous work [21] which was focused on the design of feedstock formulations based on pure nickel powder for magnetoelastic purposes. Feedstocks were developed with different percentage by volume of nickel powder and thermoplastic binder. Homogeneity of mixtures was ensured through the evolution of torque measurements. Rheological characterization of formulations led to an optimal powder loading of 50 vol.%. The flow behaviour of the prepared feedstock was found to be suitable for MIM.

The same feedstock has been used in this study to fabricate injection-moulded specimens. Commercial high pure nickel powder was supplied by Goodfellow and was produced by carbonyl reaction. The particles have a rounded shape and the particle size distribution is very narrow (between 3 and 7 μm). A carbon content of 0.07 wt.% was measured using an elemental analyser LECO CS-230, and an absolute density of 8.87 g/cm^3 by means of a helium pycnometer Micromeritics AccuPyc II 1340.

A multicomponent binder system composed of High Density Polyethylene (HDPE) and Paraffin Wax (PW) for improving flow characteristics was used. The nickel powder content was 50 vol.% whereas the remaining 50 vol.% was completed with a volume ratio of HDPE:PW = 1:1.

A ThermoHaake Rheocord 252p screw extruder was employed to produce a sufficient amount of feedstock in order to be injected. The temperature profile along the barrel was 170/175/180 °C. The feedstock was extruded twice to guarantee a high homogeneity, the first time at 15 r.p.m. and the second at 35 r.p.m. It was granulated to feed into the injection moulding machine.

The rheological characterization was carried out in order to subject the feedstock to the conditions of temperature and shear rate that would occur during the injection process in order to identify whether the mixture was suitable for this purpose. The behaviour of the mixture was determined using a capillary rheometer Dynisco LCR, where the mixture was passed through a capillary with an L/D ratio of 30 at different temperatures (160 °C, 170 °C, 180 °C and 190 °C). Ten minutes was considered enough to reach thermal equilibrium after charging the barrel.

Direct MIM injections with the above mentioned feedstock were carried out in an Arburg Allrounder 270S injection machine (Arburg GmbH, Lossburg, Germany) with 900 bar injection pressure using a cylindrical mould (6 mm in diameter and 66 mm in length). The sample is a slender rod because this geometry ensures the characterization of several magnetoelastic effects, such as magnetostriction, ΔE-effect and magnetomechanical damping, among others.

Thermal debinding was performed under nitrogen atmosphere. Different cycles were used until the optimum thermal debinding was reached. Then, the samples were sintered in a temperature range of 1300–1360 °C for different holding times (between 1 and to 20 h). A protective atmosphere of N_2–H_2 in a conventional electric furnace was used in the sintering process.

Mechanical, compositional and microstructural characterizations of the sintered samples were carried out. Densities of the sintered parts were measured via Archimedes' method using the test standard ISO 2738:1999 [22]. Vickers microhardness (VH) measurements were carried out using a microdurometer Future Tech using the test standard ISO 4498:2010 [23]. Impurities of the samples, such us C, were measured by means of an elemental analyser LECO CS-230. X-ray diffraction (XRD) and optical microscopy analyses were conducted using a diffractometer Philips X'Pert MPD PW3040 and a Leica DR-IRM optical microscope, respectively.

Finally, magnetoelastic characterization of the slender rods was obtained in terms of the ΔE-effect. The required experimental system was based on subjecting the specimens to free longitudinal vibration while they remain within different magnetic fields from 0 to 2000 Oe, and measuring their vibration by Laser Doppler Vibrometry [24].

3. Results and discussion

3.1. Capillary rheology

Capillary rheology was used to determine the rheological behaviour of the feedstock. Fig. 1 shows the dependence of viscosity on the shear

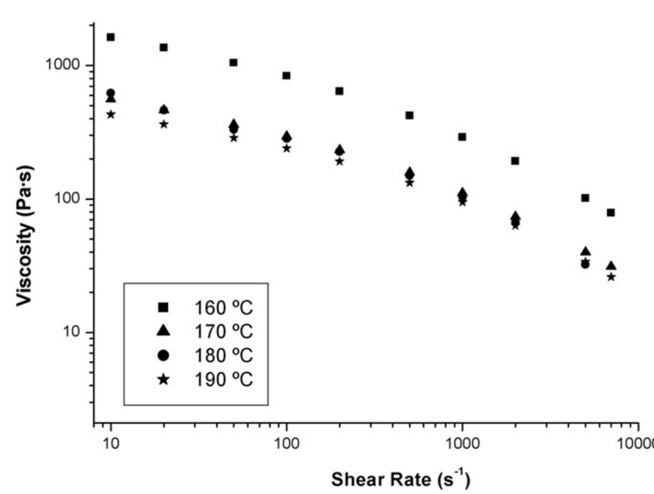

Fig. 1. Dependence of feedstock viscosity on shear rate for different temperatures (160 °C, 170 °C, 180 °C and 190 °C).