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Characterization and Heat Diffusion Characteristics of Spark Plasma Sintered Ni–50%Fe Sintered Alloy

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Abstract

The research findings on the thermal diffusivity, a(T), characteristics of Ni –50%Fe binary alloys produced by ball–milling and Spark Plasma Sintering (SPS) powder processing techniques are reported in this study. The laser flash analysis method was used to evaluate a(T). Graphite coated Ni –50%Fe specimens (cubes) with a perimeter of 4 cm and height of maximum 4 mm were analyzed at temperature range of 50 ~ 400 oC. The initial alloy powder constituents were fcc Ni and bcc Fe; milled together in a planetary ball mill and formed a bcc FeNi solid solution phase that endured throughout the sintering stage, and contained in the microstructure of the finished product. The rate of heat diffusion in metallic Ni – 50%Fe compacts sintered at 800 oC is strongly affected by the intensity of milling/formation of new solid solutions. This value is small for long milling duration feedstock powders, even though the results from both powder feedstock milling apparatus seems to display a declining trend for all Ni – 50%Fe compacts sintered at 800 oC.

Keywords: Thermal diffusivity, Laser flash analysis, Spark plasma sintering, milling, Ni–50%Fe compacts;

1. Introduction

A number of research studies have been implemented on the development and production of components using nano-engineered materials for diverse applications in the last decade. However, it requires a deep understanding of the methods for producing components with nanocrystalline structures and the multidimensional approach to performance evaluation of these parts. This entails understanding the following: suitable materials, compositional formulation methods, production process factors and the materials' responses to applied forces under processing. In addition, the right characterization techniques to understudy the structural (macro, micro, nano), chemical, and thermo-mechanical behaviours under the processing and application environments [1-5].

Nickel based alloys are used for a variety of engineering applications, including aircraft engine components with accomplishing properties, such as high strength at elevated temperatures, corrosion resistance, fatigue and toughness [6]. For such alloys to be used for higher temperature applications, alloying element modification, heat treatment and processing route optimization steps are continually performed to further enhance the properties [7-9]. Improved mechanical and physical properties are strongly dependent upon the morphologies, type and distribution of the second phases. This is in turn a function of alloy composition and it is in majority of metal working techniques which involve the melting and the cooling rate [10-11]. This has made it necessary to dedicate material processing techniques to precision capabilities. In this regard, powder processing technologies are taking over the market as a solution to low cost manufacturing, tailored service properties and increased serviceability [12-13].

The evaluation of thermal properties of new materials is quite important. For several engineering applications in microscopic or macroscopic structures, the knowledge of their capabilities to dissipate heat is paramount. Research into thermal diffusivity of materials has been and still remains a huge challenge for materials used in ambient temperatures. It is renown that thermal diffusivity measurements take important part in the material science. Thermal diffusivity measures the heat transfer rate of a material from the hot side to the cold side. It is obtained from the ratio of thermal conductivity to the product of density and specific heat capacity at constant pressure as presented in (1).

$$\alpha = \{k/(\rho C_p)\} \quad (1)$$

where $\boldsymbol{k}$ is thermal conductivity (W/(m·K)), $\boldsymbol{\rho}$ is density (kg/m³), $\boldsymbol{C_p}$ is specific heat capacity (J/(kg·K)), or ρC_p denotes the volumetric heat capacity (J/(m^3·K)) [14].

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Thermal diffusivity is the ratio of the time ***t*** derivative of temperature ***T*** to its curvature as given by the heat equation (2). [15]

$$\partial T/\partial t = \propto \nabla^2 T \quad (2)$$

Thermal diffusivity is among the basic parameters for Infra-Red Non-Destructive Testing [IRNDT] using flash heating which gives the possibility to detect defects as well as the depth of their occurrence [16-18]. From the material science point of view, three factors play an instrumental role in determining thermal diffusivity using the flash method. These include measurement of the stabilized temperature of the sample immediately before the laser short onto its front surface; temperature variation at its edges [18]. Flash methods for determination of thermal diffusivity require knowledge of the exact thickness of the studied objects or surface emissivity and absorptivity for correct determination of temperature distribution and light absorption. In this method, a flash-light source and Infra Red (IR) camera are installed in front of the studied object and a short flash irradiation of the object provides an instantaneous source of heat on the surface [17]. The rate at which the heat travels from one surface to the other front is calculated.

The setup was experimented on a Ni-50%Fe binary systems produced by ball-milling and the subsequent spark plasma sintering using hot furnace (model HHPD – 25 from FCT Germany) at constant temperature of 800^{o}C. Two different planetary ball-milling equipment (PM 100 CM and PM 400 MA) were used to prepare the feedstock powders under the same speed, milling duration and charge input in wet conditions. Ball-milling of metal powders by mechanical forces results in size reduction, particle shape deformation, cold welding and inter- atomic diffusion [19-20]. SPS is amongst the latest development in powder processing technologies which has been receiving attention for rapid powder consolidation of highly dense compacts at low temperatures and minimal coarsening of grains [21]. Milling of metal powders is increasingly receiving attention as a means for kinematic coagulating of metal powders of improved characteristics including shape and structure [22-23]. The structural characteristics of the system were discussed through the X-Ray Diffraction (XRD) analytical technique using the XRD Empyrean Model with X'Pert HighScore analytical software. The thermal diffusivity measurements on the Ni-50%Fe compacts sintered at 800^{o}C are being investigated. This work is a part of large investigation on the NiFe alloy by the Centre of NanoEngineering and Tribocorrosion, University of Johannesburg already reported and published in public domain [24-27].

2. Experimental

2.1. Starting materials

The starting materials used are Ni powder (particle size <325 mesh; purity 99.9%; Alfa Aesar) and Fe powder (particle size <325 mesh; purity 99.9%; Weartech (Pty) Ltd). The morphology of the starting powders was determined using SEM (field emission gun scanning electron microscope equipped with EDS) and are presented in Figure 1, (A.1- A.3, B.1-B.3) The phases present in the milled/sintered samples were characterized by X – ray diffraction (XRD) using a PANanalytical Empyrean model with CuKα radiation and analyzed using Highscore Plus software and the lattice parameter were calculated using the Sherrer equation in (3):

$$Crystallite\ size\ = k\lambda/B\cos\theta \quad (3)$$

Where k is the Scherrer constant, λ the wavelength and B is the full width at half maximum FWHM (radians) and θ is diffraction angle

2.2. Ni–50%Fe powder systems

Figure 2 shows the morphology of individual Ni (A.1-A.3) and Fe (B.1-B.3) milled for 24 hours in a PM 400 MA apparatus. A low and high energy planetary ball mill, Fritsch Pulversette 6 (PM 100 CM and PM 400 MA) were used during wet mixing of Nickel and Iron powders in the ratio 1:1, using ethanol as the process control agent. The mixing vessel used inside the planetary ball mill was made from stainless steel with a capacity of 250 ml. The mixing speed was kept at 300 rpm, while using Steel balls of 2.5 mm in diameters as milling media. Mixed powders of unmilled Ni – 50%Fe powder combination (Figure 3, (A.1-A.2)) and mixed powders of 24 hous milled Ni – 50%Fe powder combination (Figure 3, (B.1-B.2)) were charged into two different planetary ball milling apparatus (PM 100 CM and PM 400 MA) in preparation for SPS feedstock. In Figure 4, (A.1-A.2) low energy milled (PM 100 CM) powder product and Figure 4, (B.1-B.2) high energy milled (PM 400 MA) product are presented. The two powder products were prepared for use as feedstock material for the spark plasma sintering furnace.

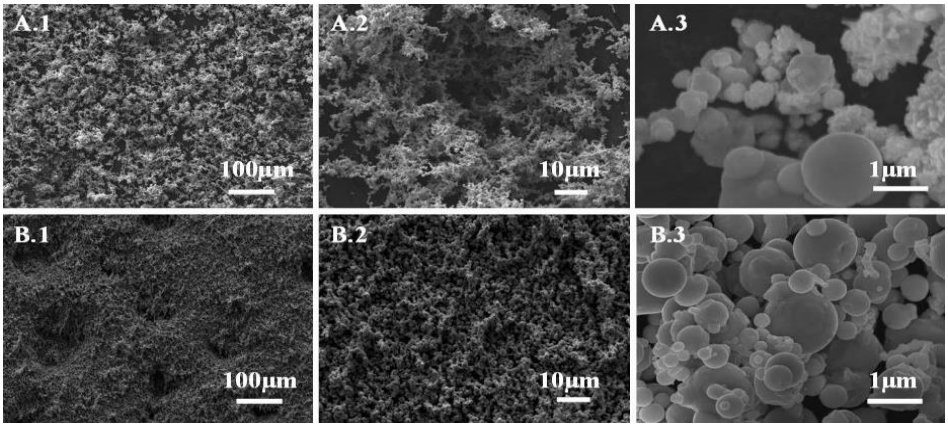

Figure 1. Morphology of as - received powders (A - Nickel and B - Iron)

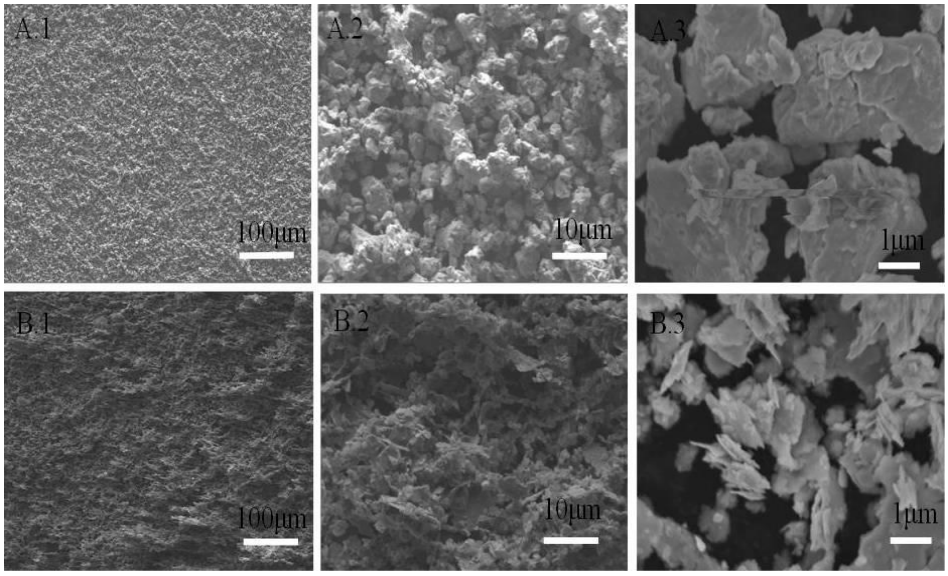

Figure 2. Morphology of individual Ni (A) and Fe (B) milled for 24 hours in a PM 400 MA apparatus

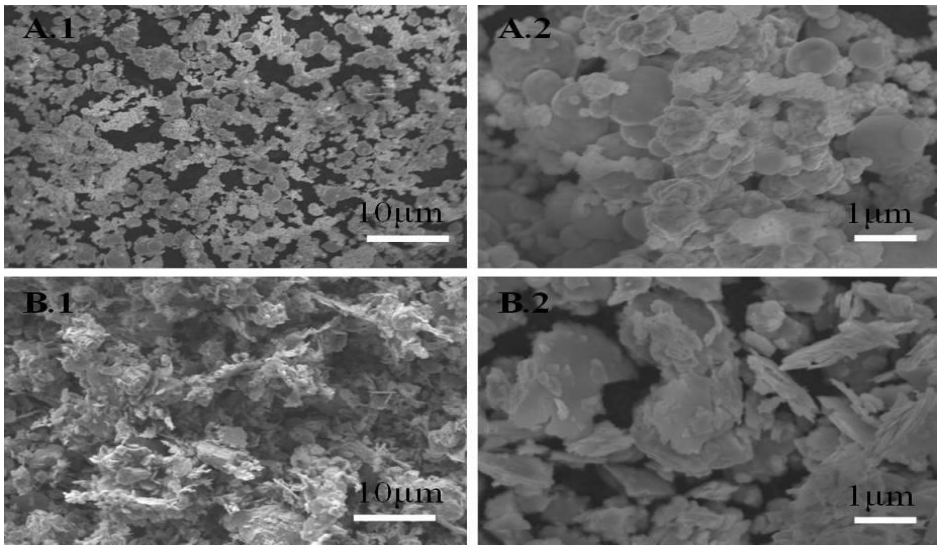

Figure 3. Mixed unmilled Ni - 50%Fe powder combination (A) and 24 hours milled then mixed Ni - 50%Fe powders (B)

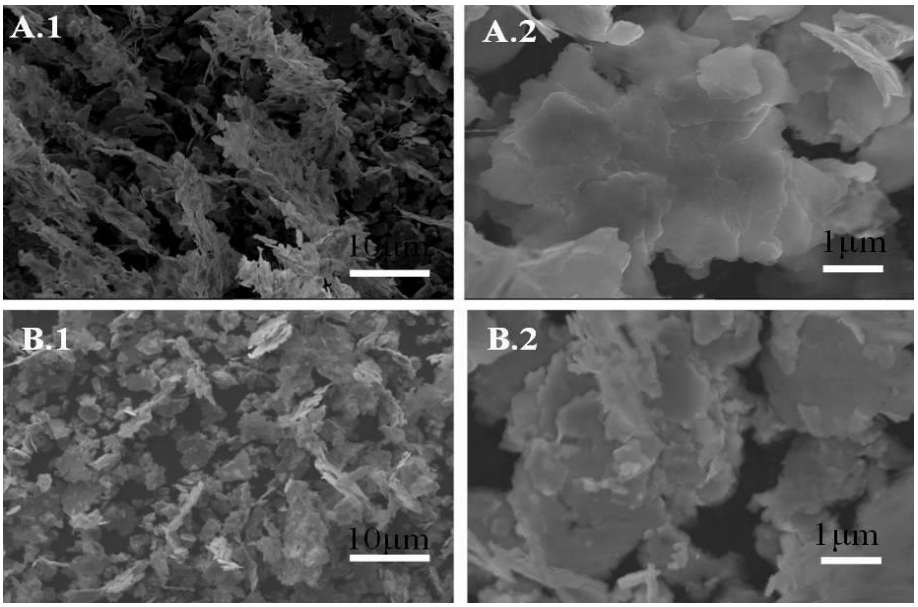

Figure 4. 20 hours milled Ni - 50%Fe powder feedstock (A - PM 100 CM and B - PM 400 MA)

2.3. Sintering

The milled feedstock powders were poured into a graphite die with diameter 30 mm and sintered by the spark plasma sintering system (model HHPD – 25 from FCT Germany) at constant temperature and heating rate in a vacuum. The sintering temperature was measured by an optical pyrometer located 3 mm from the top of the sample surface. The furnace was heated to 800^{o}C at 150^{o}C/min and held at this temperature under a pressure of 45 MPa for 10 minutes. Cylindrical specimens of 30 mm diameter and approximate height of 5 mm were produced. Density measurements of the sintered specimens were done using the Archimedes method. Then the samples were sectioned and ground for metallographic examination according to standard procedures.

2.4. Thermal diffusivity measurement

The investigation of the thermal diffusivity of Ni – 50%Fe sintered alloys was conducted according to the ASTM E1461 – 13, E2585 - 09(2015) standard practices. The samples was coated with graphite in order to enhance the absorption of laser energy and the resulting signal– to–noise ratio especially when surface is reflective. For coating, all samples are cleaned with a suitable solvent and laid side by side then sprayed at approximately 25-30 cm away from the samples and a 5 μm thickness layer is introduced across the entire surface area of the sample. The graphite furnace measures up to 2000 ^{o}C and consists of a protective tube which separates furnace from the sample chamber. The graphite material requires that the furnace chamber be always purged with an inert gas.

Ni – 50%Fe sintered specimen were cut to square shape of 4 cm perimeter by 4 mm thickness for measurement of thermal diffusivity by laser flash analysis (LFA) using LFA 427 Microflash, (Netzsch, Germany). The LFA beam is absorbed by a thin film on the front of the test piece. The layer (sprayed graphite) serves to prevent reflection of the laser beam from the surface of the Ni – 50%Fe samples. Since the temperature separation (difference) inside the test piece depends only on the thermal diffusivity of the material. The dynamic test was performed form 50 oC to 400 oC at a heating rate of 5 K/min and the Thermal diffusivity measurements were taken at 50oC temperature interval and constant time.

3. Results and Discussions

3.1. Morphology of as - received powders and Ni – 50%Fe powder systems

Figure 1 shows the morphology of as - received powders (A.1-A.3) - Nickel and Iron (B.1-B3). Spherical shaped particles for both Ni and Fe were observed, with Ni having the highest degree of agglomeration.

A 24-hour milled individual Ni and Fe powder morphologies are presented in Figure 2. Ni continues to display a higher degree of agglomeration (Figure 2, A.2 & B.2) as compared to Fe, and because of the malleable characteristic of the iron powders, the milled powder products are of very thin sheets.

The goal of the mechanical milling of metal powders in the preparation for Spark Plasma Sintering was to attain a sufficiently fine microstructure, with minimized chemical interaction between elements by continuous abrasion of the powder mixture [22].

The morphology of individual Ni (A) and Fe (B) milled for 24 hours in a PM 400 MA apparatus is shown in Figure 2 while Figure 3 shows the mixed unmilled Ni - 50%Fe powder combination (A) and 24 hours milled then mixed Ni - 50%Fe powders (B). The products of 20 hours milled Ni - 50%Fe powder feedstock (A - PM 100 CM and B - PM 400 MA) are illustrated in Figure 4.

3.2. X – Ray Diffraction analysis

Phase analysis of raw nickel and iron powders, as well as 24 hours milled Ni and Fe powders are presented in Figure 5 (A – D) respectively. Calculated values of the average crystallite size of powders and powder systems are presented in Table 1. Phase analysis for 20 hours milled Ni – 50%Fe powder combination for different milling apparatus, together with the sintered feedstock produced at 800^{o}C for different mill product are also presented in Figure 6 (A & B).

As anticipated, milling of metal powders in high energy planetary ball mills greatly alters the size characteristics of powders , the broadening of X –ray diffraction peaks is indicative of size reduction (Figure 5 B & D). The overall decrease of crystallite size of pure Ni powders is over 90% (from 68.89 to 6.36 nm) and the overall decrease in crystallite for pure Fe powder is on over 60 % of the initial crystallite size. This results are indication that Ni, which has a characteristic ductility is severly fragmented during milling as compared to malleable Fe powders, which by virtue of visual observation of Figure 2, (B.3) displays rod – like powder particles when milled for 24 hours in PM 400 MA apparatus. During milling of Ni and Fe powders, the action of Ni atoms entering the Fe lattice is imminent because of the difference in atomic packing factor, and thus, in every physical encounter, a FeNi intermediate solid solution is formed. The solid solution phase is contained in the 20 hours milled powders (Figure 6, A) for powders milled from both milling machines, even though the high energy milled Ni – 50%Fe powder combination displays a greater degree of peak broadening. This is validated by the small average crystallite size (7.01 nm) of the powder. In addition to drastic crystallite size reduction capabilities of the PM 400 MA milling apparatus, even element distribution is achieved. The phase analysis of the sintered powders from both milling apparatus at 800^{o}C are presented in Figure 6 (B). It was observed that the FeNi solid solution endured throughout the sintering stage and as contained in the structural definition of the finished product.

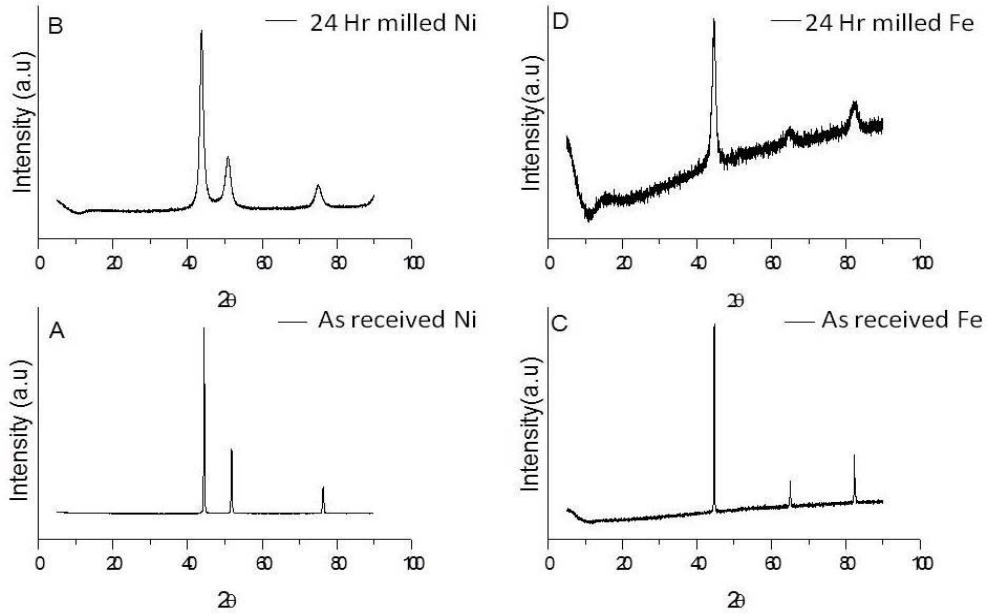

Figure 5. Phase analysis of raw powders: A & C as - received Nickel and Iron powders; B & D - 24 hours milled (PM 400 MA) Nickel and Iron powders

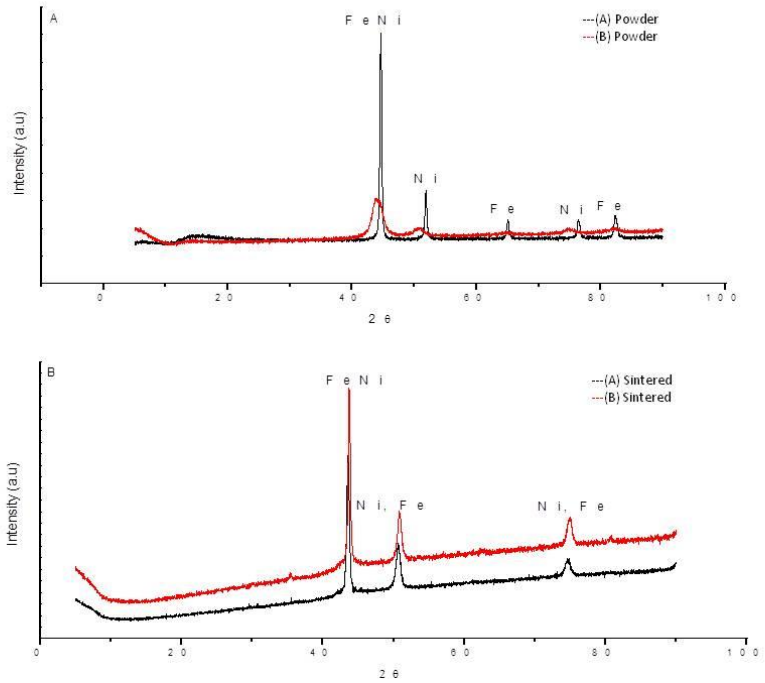

Figure 6. Phase characteristics of Ni - 50%Fe powder systems (A) and Ni - 50%Fe compacts sintered at 800 ^{o}C (B)

Table 1. Crystallite measurements of raw powders and Ni - 50%Fe powder systems

Description	Average crystallite size (mesh size)
As – received Ni	68.89
24 hours PM 400 MA milled	6.36
As – received Fe	271.07
24 hours milled Fe	101.78
20 hours PM 100 CM milled Ni – 50%Fe **(A)**	14. 74
20 hours PM 400 MA milled Ni – 50%Fe **(B)**	7.01
Ni – 50%Fe 800^{o}C sintered **(A)**	48.74
Ni – 50%Fe 800^{o}C sintered **(B)**	72.19

3.3. Heat diffusion characteristics of Ni – 50%Fe sintered compacts.

Thermal diffusivity data plots and values for sintered specimen of A and B at temperature range of 50 to 400°C are presented in Figure 7, and 800°C in Figure 8 and Table 2 respectively. The packaging densities of sintered A and B are 8.213 and 7.562 g/cm^3 respectively.

Thermal diffusivity of sintered Ni – 50%Fe compact prepared from P 100 CM feedstock powder is obviously larger than that of Ni – 50%Fe compact prepared from PM 400 MA feedstock powder at the same temperature. The sintered $_A$ decreases linearly (from 5.23 mm^2/s) with increasing temperature. For the two specimen, the increase in the milling intensity leads to more difficulties for the heat diffusion, as can be seen in Figure 8, the total duration of milling of metal powders before sintering is 44 hours and the value of thermal diffusivity (2.796 mm^2/s) at 50°C is small as compared to sintered A specimen at the same analysis temperature. According to Terpilowski et al [28], diffisivity values of iron – nickel binary alloys is likely to spike when approaching the curie temperature of the alloy beacause at this temperature the material lose their permanent magnetic properties to be replaced by induced magnetism. The thermal diffusivity of the sintered high energy milled powder feedstock follows a singular trajectory with the low energy milled sintered powder feedstock.

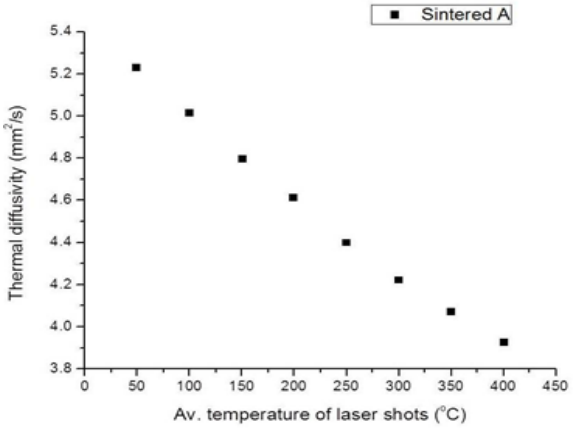

Figure 7. Thermal diffusivity measurements of Ni - 50%Fe sintered compacts prepared from low energy mill (PM 100 CM) powder feedstock

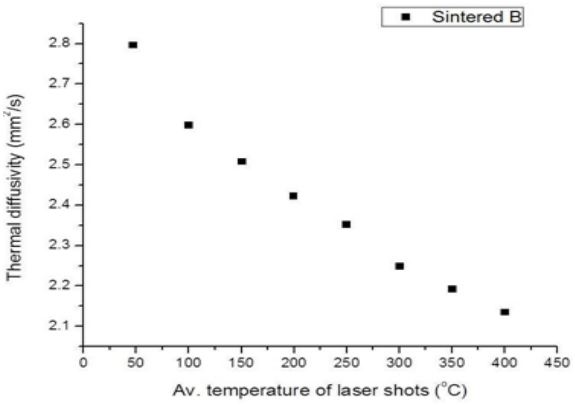

Figure 8. Thermal diffusivity measurements of Ni - 50%Fe sintered compacts prepared from high energy mill (PM 400 MA) powder feedstock.

Table 2. Thermal diffusivity values of Ni - 50%Fe sintered specimen (A & B) at different temperatures

Sintered A		**Sintered B**	
Mean Temperature (°C)	Thermal diffusivity (mm^2/s)	Mean temperature (°C)	Thermal diffusivity (mm^2/s)
49.6	5.23	47.1	2.796
100.1	5.015	100.2	2.598
150.7	4.796	150.8	2.508
199.5	4.613	199.4	2.422
249.8	4.399	249.9	2.351
300.1	4.222	300.2	2.248
350.3	4.071	350.4	2.192
400.4	3.927	400.5	2.135

4. Conclusions

Ni – 50%Fe binary systems prepared from two different milling apparatus (PM 100 CM and PM 400 MA) produce powders of a singular structural characteristics when powders are milled for the same time (20 hours). There are spectacular degree of size reductions marginally large for powders produced from high energy milling apparatus. The high energy milled powder feedstock results in low packaging density sintered specimen as compared to PM 100 CM powder feedstock when sintered at the same temperature of 800°C. The heat diffusion rate in Ni – 50%Fe sintered compacts is strongly reduced when powders are milled for longer times before sintering. Thus the results obtained from this evaluation of thermal diffusion properties of the developed NiFeCo alloy is very important for its several applications at microscopic or macroscopic scales, especially where the understanding of their heat dissipation capabilities is vital.

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