The study of microhardness of powder metallurgy fabricated Fe-Cu alloy using vickers indenter

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Received: 29 March 2016, Revised: 30 September 2016 and Accepted: 15 December 2016

DOI: 10.5185/amp.2017/411 www.vbripress.com/amp

Abstract

In the present study, micro hardness values of Iron-Copper based alloys with different compositions have been obtained by using Vickers indenter. The samples Fe-5Cu-1Sn-7.5BN (wt. %), Fe-5Cu-1.5Sn-5BN (wt. %) and Fe-5Cu-2.5Sn-2.5BN (wt. %) were prepared using Powder metallurgy technique. The elemental powder mixture was mixed for 2 hours, and compacted at a pressure of 500Mpa, and then was sintered in dry hydrogen atmosphere at a temperature of 900°C for 50 minutes time. Vickers hardness values were obtained under the loads of 0.01 kg to 0.3 kg. The material with 2.5 wt. % of Sn exhibited the highest value of the hardness. The material with higher hardness shows better mechanical and tribological properties and can be used for various applications such as gears, bearings, connecting rods cams etc. Copyright © 2017 VBRI Press.

Keywords: Vickers hardness, micro indentation, powder metallurgy, sintering, iron-copper alloy.

Introduction

Change is perpetual. Since earlier times industries have always met a drastic change in which classical materials have been replaced by the latest and advanced ones to meet the demands of the current materialistic world whether in the field of aeronautics, nuclear science, terrestrial or maritime transportation, etc. Nowadays industries are based upon the substitution of classical materials with more advanced ones, whose properties maintain higher altitudes of success. The latest materials and advanced technologies fix up all the limitations faced in the usage of the traditional materials in respect of the limited strength and resistance values [1].

Powder metallurgy, a beautiful alternative to produce metal products with advanced technologies from metal powders (sometimes mixed with non-metallic powders) by application of pressure and sintering. Powder metallurgy technique has become increasingly interesting for manufacturers of engineering parts due to its advantageous qualities which include high productivity, minimum consumption of raw materials and energy, high efficient use of the initial metals, near net shape character and unique capability of porous material production. Application of sintered parts is expanding in automobile and other engineering industries because of both economic and technical reasons. Machine elements which are complex in shape like gears, bearings, connecting rods, Cams, etc. can be economically manufactured through the powder metallurgy processing route **[2-3]**.

In recent years, iron-based sintered materials were considerably increased, due to low cost and availability of the iron powders as well as their higher strength. The mechanical properties of the parts are strongly related to the composition of the material. The material density plays an important role for exhibiting good load bearing capacity. Therefore, it is essential to know the actual loading conditions of the part and modify the alloying and the treatment conditions of the material on the basis of these conditions.

Sintered iron based materials were not developed till last decades because of their poor corrosion resistance. Considering the low cost and availability of iron powders, the more homogeneous structures obtained by sintering, the increased specific load-carrying capacities and sliding rates, the development of the Fe base sintered alloys for mechanical applications was continuously improved. Additions such as copper, graphite, manganese, lead, phosphorous, boron, and tin to iron have been attempted, but improvement in one property was offset by decrease in other property [4-7]. To overcome weaknesses in existing alloy systems and to meet the challenging nature of newer machines, it is important to develop a modified alloy system which can succeed in dealing with these deficiencies, either partially or fully.

Using conventional powder metallurgy techniques, the present work is focused on the development of new Fe-Cu base materials with different addition elements such as tin, and Boron Nitride powders. In sintered Fe base materials copper has unique properties as an alloying element. In small amounts copper improves strength and rust resistance, and also has a rapid surface diffusion over solid iron. Tin is a key player which influences the corrosion resistance and fatigue strength. Mechanical and tribological properties, such as hardness, strength and wear resistance depends upon the (a) Sintering additives (b) type of reinforcement and (c) sintering conditions (i.e. temperature, pressure etc.) [8]. Hardness plays a significant role, in increasing the wear resistance, higher hardness means higher wear resistance and vice versa [9]. Hardness is crucial for cutting tools, wear and abrasion- resistant parts, prosthetic hip joint balls and sockets, optical lens glasses, ballistic armor, molds and dies, valves, and seals [10]. In order to understand the hardness values and wear resistance, it is essential to measure hardness of developed material. The main objective of the present study is to develop the bearing material with higher hardness to show better mechanical and tribological properties. This can be achieved by finding the appropriate wt% of the Sn material and its micro indentation at various loads and indentation time. The novelty of the current study is Iron as a base material for bearings, which has been very less studied. Iron has higher strength, readily available and low cost when compared with other materials like copper which is generally used as a bearing material. The microhardness study of the material with Tin addition has not been reported yet. The results obtained during this study reveals that the Fe-Cu base material with variation of Sn as an alloying element shows different Vickers hardness results at different loads. These results can be better used by various manufacturers to develop the bearing which can have better mechanical and tribological properties.

Experimental

Materials and manufacturing process

In this study, the Iron (Fe) powder, used as a base material, was procured from Sigma Aldrich USA, with an average particle size of $80\mu m$ (**Fig. 1**). The copper (Cu) powder with a fixed 5wt% (procured from SRL Laboratories India) is added, as it has been found to improve the strength while sintering [10].

Table 1. Experimental conditions.

Temperature	°C	900	
Holding Time	Min.	50	
Atmosphere		Dry Hydrogen	

The Tin (Sn) powder procured from Nano Shell Company USA, were varied. The final wt. % composition of the selected powder materials is shown in **Table 1**. Elemental powders were weighed to the selected proportions and mixed for 3 hours, then the mixed powders were cold compacted at 500 MPa into a rigid die by applying a pressure in single axial direction through a rigid punch, and 25mm diameter and 6mm thick samples were obtained. The green samples were placed in a tubular furnace *Nabertherm* made with a maximum temperature up to 1500° C, having a uniform heating zone and sintered at 900°C for 50 minutes. The sintering atmosphere was dry hydrogen. The samples were then furnace-cooled by switching off the power and maintaining the same flow rate of the hydrogen gas. **Fig. 2**, shows the optical microscopy of the sintered sample. It is clear from **Fig. 1** that the powder mixture is homogenous and EDS (**Fig. 1(b**)) shows the presence of all the elements of the composition.

The composition of the powder mixtures is presented in **Table 2**.

Table 2. Composition of the mixed powders (Wt%).

Sample	Iron (Fe)	Copper (Cu)	Tin (Sn)	Boron Nitride (BN)
А	Bal.	5	1	2.5
В	Bal.	5	1.5	5
C	Bal.	5	2.5	7.5





Fig. 1. (a) SEM and (b) EDS of the mixed powder.

Characterization

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) studies were carried out to study microstructure of developed samples. Typical results of SEM with EDS are shown in **Fig. 3(a-d)**.



Fig. 2. Optical microscopy of the sintered sample (a) $100 \mu m$ and (b) $10 \mu m.$

It is obvious from **Fig. 1**, that these microstructures emphasize a relatively uniform distribution of the phases in the ferrite matrix, with distinctive boundaries. EDS analysis of the Fe-Cu alloy was carried out at five different spots marked as spectrum 1-4 which shows the presence of all the constituents as shown in **Fig. 3(a-d)**. It is evident from EDS analysis of these four spots that the mixed composition is evenly distributed over the surface of Fe-Cu alloy.



Fig. 3. SEM and EDS of Fe-Cu alloy (a, b, c, d shows the EDS at point 1-4).

Microhardness tests

Hardness tests were performed on polished surfaces (mirror like finish) using Vickers indenter. Vickers hardness (HV) tests were performed on [*INNOVATEST Falcon 500*] micro hardness tester. The indentation was observed under 10 and 40 magnifications. HV were measured to study influence of indentation load and time on the hardness values. The indentation load was varied from 0.01kg to 0.3 kg and indentation time was varied from 1 seconds to 10 seconds. In order to understand the influence of indentation tests were performed on selected Fe-Cu base samples. Each indentation of tests was repeated 3 to 4 times for better repeatability.

Results and discussion

Hardness values (HV) of Fe-Cu base alloy with different compositions against indentation load and indentation time are shown in **Fig. 4** and **Fig. 5**. The influence of indentation load (0.01kg -0.3 kg) on HV of iron base alloy with 1 wt%, 1.5 wt%, and 2.5 wt% Sn is shown in **Fig. 4(a)**. As can be seen in **Fig. 4(a)**, the hardness decreases with the increase in load in iron base samples containing 1 wt%, 1.5 wt%, and 2.5 wt% Sn. However, higher hardness values were obtained in sample C containing 2.5 wt% Sn as compared with other samples A and B containing 1 and 1.5 wt% Sn respectively.



Fig. 4. (a) Vickers Hardness (HV) verses load for samples A, B, and C containing 1, 1.5, and 2.5 wt% Sn; Vickers Hardness (HV) verses indentation time at: (b) 0.01 kgs constant load varying indentation time from 1-5 seconds and (c) 0.01 kgs constant load varying indentation time from 6-10 seconds.

This behavior is attributed to the formation of Cu-Sn, Fe-Cu, and Fe-Sn compounds in large amounts at higher temperatures as the Sn content increases. Liquid Sn has substantial solubility in the solid iron, which aids in the formation of liquid phase during sintering, which on solidification produces some strengthening phases such as Fe-Sn, Fe-Cu, and Cu-Sn.

The indentation time tests were carried out to determine the influence of indentation time on hardness value (HV). These indentation tests were performed on iron base samples with different compositions. The influence of indentation time (1-5 and 6-10 seconds) on HV of sample A, B, and C containing 1 wt%, 1.5 wt% and 2,5 wt.% Sn at constant loads of 0.01 kg is shown in **Fig. 4(b&c)**. **Fig. 5(a&b)** indicate the influence of indentation time (1-5 and 6-10 seconds) on HV of sample A, B, and C containing 1 wt%, 1.5 wt% and 2,5 wt.% Sn at constant loads of 0.05 kgs.



Fig. 5. Vickers Hardness (HV) verses indentation time: (a) at 0.05 kgs constant load varying indentation time from 1-5 seconds; (b) at 0.05 kgs constant load varying indentation time from 6-10 seconds; (c) at 0.3 kgs constant load varying indentation time from 1-5 seconds; (d) at 0.3 kgs constant load varying indentation time from 6-10 seconds.

Whereas, the influence of indentation time (1-5 and 6-10 seconds) on HV of sample A, B, and C containing 1 wt%, 1.5 wt% and 2,5 wt.% Sn at constant loads of 0.3 kgs is shown in **Fig. 5(c&d)**. As can be seen in **Fig. 4(b&c)** to **Fig. 5(a-d)**, the hardness HV decreases with the increase in indentation time (2 second -10 seconds), however as seen the **Fig. 5(a-d)**, at 0.05 kg and 0.3 kg, there is less decrease in the HV value with increase in indentation time from (2 second – 10 seconds). This indicates that with increase in indentation time at higher loads there is a lower value of hardness in the material.

As mentioned in previous section that indentation (Vickers) were observed under optical microscope at 10 and 40 magnifications. Indentation images on the surface of developed materials are shown in **Fig. 6(a-c)**. **Fig. 6** indicates the indentation image of Vickers indenter at 0.1 kg load at indentation time of 10 seconds. **Fig. 6(d)** shows the cracked image of the sample having indentation load greater than 0.3 kgs.



Fig. 6. (a-c) Indentation Images of Samples A, B, and C at indentation load of 0.1 kg for 10 Sec; (d) Indentation Image showing cracks above 0.3 kg load.

Conclusion

Iron-Copper alloy with different compositions were developed using powder metallurgy technique. The Microhardness values (HV) of Iron-copper alloys were studied using Vickers indentation methods. The hardness decreases with the increase in load in iron base samples containing 1, 1.5, and 2.5 (wt. %) of Sn. Higher hardness values were obtained in the composition of 2.5 wt. % of Sn, as compared with other compositions of 1 and 1.5 wt. % of Sn. The increased percentage of Sn aids in the formation of liquid phase sintering, which on solidification produces some strengthening phases such as Fe-Sn, Fe-Cu, and Cu-Sn. The highest value of hardness 462-HV0.01 was obtained in composition containing 2.5 wt% of Sn, the highest value of hardness is better than the existing iron base alloys, which directly increases the wear resistance of material. Therefore, such materials can be used for various applications importantly in plain bearings, gears and various automobile and machinery equipment's.

Acknowledgements

The authors wish to acknowledge the kind support by Prof. D. Sarkar, National Institute of Technology Rourkela, India for helping me in sample preparation and High temperature tribology laboratory, National Institute of Technology, Srinagar, India where the work was performed.

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