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Chapter 28
Correlation Between Mechanical Properties and Microstructure of Fe-Ti-Zn Alloys Fabricated by Powder Metallurgy

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Abstract Two Iron-based alloys namely, 93Fe-5Ti-2Zn and for 88Fe-10Ti-2Zn, Specimens were prepared using powder metallurgy techniques. XRD results on both alloys showed the presence of intermetallic phase in 88Fe-10Ti-2Zn. The hardness of 88Fe-10Ti-2Zn samples was higher than that of 93Fe-5Ti-2Zn sample. Impact value for Sample 1 (93Fe-5Ti-2Zn) was 66 J and for Sample 2 (88Fe-10Ti-2Zn) was 73 J. Mechanical property values were correlated to the XRD and microstructures obtained.

28.1 Introduction

Iron melts at 1538 °C and Titanium at 1668 °C. This being the case, manufacturing an alloy of Iron and Titanium with minor alloying addition of Zn becomes very cumbersome and energy consuming if it is done by the normal casting process. Powder metallurgy is a route which has been followed for the last 30–40 years in order to manufacture such high melting alloys. While mechanical properties obtained are comparable to alloys manufactured by casting techniques, reduction in porosity is an added advantage if powder metallurgy is used. Titanium was chosen as the major alloying element in order to reduce weight of the alloy and Zinc was chosen

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as the minor alloying element in order to reduce corrosion resistance. Many high melting alloys have been fabricated by using Powder Metallurgy, most commonly Aluminium and Titanium based refractory alloys and Nickel-based Superalloys. Fe-35 Mn alloys have been manufactured for use in stents [1]. Iron-Copper Tin Lead alloys have been fabricated using P/M for use in self-lubricating bearings [2, 3]. On studying the Iron-Titanium phase diagram, it is found that up to 15% Titanium, we have ferrite at higher temperatures. Since Ferrite is soft and not so strong, it is not possible to strengthen Iron-Titanium alloys using casing techniques [4]. Hence, it is more important that powder metallurgy techniques are made use of. Iron-Titanium system which is known to show the presence of intermetallics, namely Fe-Ti and Fe_2Ti.

28.2 Experimental Work

28.2.1 Manufacture of Alloy Using Powder Metallurgy

In this process, the final component is made from a mixture of powders. Usually, metal removal rates can be drastically reduced as the process is a near net manufacturing process. Thus it is clear that yield will be more and losses are reduced, thereby reducing costs. But, on the other hand, the high volume to the surface area of the powders renders the powders more reactive to the atmosphere. Highly reactive powders like Titanium and Iron are more prone to oxidation.

Many special products are possible with powder metallurgy technology. A non exhaustive list includes Al_2O_3 whiskers coated with very thin oxide layers for improved refraction; iron compacts with Al_2O_3 coatings for improved high temperature creep strength; light bulb filaments made with powder technology; linings for friction brakes; metal glasses for high-strength films and ribbons; heat shields for spacecraft reentry into Earth's atmosphere; electrical contacts for handling large current flows; magnets; microwave ferrites; filters for gases; and bearings which can be infiltrated with lubricants.

One should be extra careful of metal powders and adequate safety precautions should be taken, especially when bulk powders are handled. This is because in the finely divided form many powders pose health hazards. The same powder which may be benign in bulk form can be dangerous in fine powder form (Fig. 28.1).

Powder metallurgy consists of the following process.

- Mixing of powder
- Compacting
- Sintering
- Secondary Finishing.

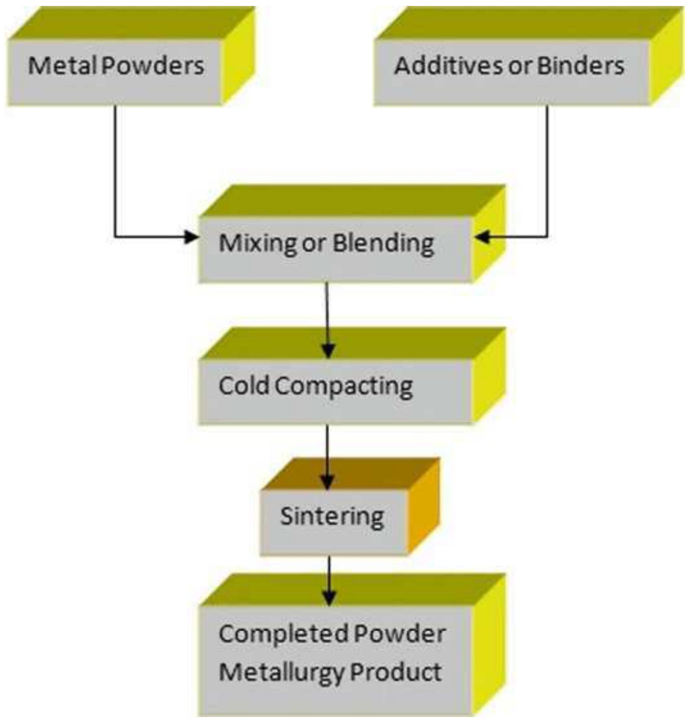

Fig. 28.1 Flow chart indicating the powder metallurgy process

28.2.2 Mixing of Powders

Mixing of the powders is the most important step since proper mixing will ensure uniform distribution of the constituents. If the constituents are not mixed properly, there is a possibility of obtaining non-uniform mechanical properties, while mixing and compacting, lubricants like stearic acid added to help in lubrication. The alloy is mixed under two different proportions and compacted. The two alloys are tested for various mechanical properties and microstructure and were compared.

The powders of iron, zinc and titanium are mixed in the planetary ball miller. Ball mills are of different types like horizontal, vertical, tumbler mill, cement mill, etc. Here, a laboratory scale model has been used. The number of balls and the number of hours of milling are important variables. Normally, research is performed varying the number of hours of milling as 5, 10, 15 and 20 h. In this case, 8 h of milling has been used. 0–2 wt% stearic acid or magnesium stearate have been used in literature to mill Titanium-based powders. This internal lubricant improves compressibility [5]. A lubricant such as stearic acid, stearin, metallic stearates or other organic compound of a waxy nature is added to the mixture. The mixture is milled in the planetary ball mill for about 8 h. Finally, the mixture is taken out. Graphite has also been added as a lubricant during Powder Metallurgy manufacture of alloys [6]. Stearic acid and Molybdenum disulfide are some of the other lubricants commonly used in powder metallurgy. The mixed powder may be injected into a die or a press.

28.2.3 Compacting the Mixture

The mixture of iron, titanium and zinc powders obtained from the planetary mill is then compacted to solid of 40 × 15 × 10 mm rectangle. First, the die cavity is filled with the powder mixture. The die is pressed in the hydraulic pallet press. The pressure of about 650 N/mm^2 is applied on the die. Then the compacted powder mixture is ejected out of the die and left for some time. Generally, it is enough if cold compaction is performed [7]. However, in case where very high accuracy and surface finish is required, for example in defence applications hot compaction is resorted to attain high accuracy and surface finish [8]. Hot compaction is sometimes referred to as Hipping or Hot isostatic pressing [9]. The word isostatic means applying equal pressure from all directions. This ensures uniformity of properties.

28.2.4 Sintering

Sintering can be considered to proceed in three stages. During the first, neck growth proceeds rapidly but powder particles remain discrete. During the second, most densification occurs, the structure recrystallizes and particles diffuse into each other. During the third, isolated pores tend to become spheroidal and densification continues at a much lower rate. The words ‘solid state’ in solid state sintering simply refers to the state the material is in when it bonds, solid meaning the material was not turned molten to bond together as alloys are formed [11].

One recently developed technique for high-speed sintering involves passing a high electric current through a powder to preferentially heat the asperities. Most of the energy serves to melt that portion of the compact where migration is desirable for densification; comparatively little energy is absorbed by the bulk materials and forming machinery. Naturally, this technique is not applicable to electrically insulating powders.

Titanium and Iron are both highly reactive with Iron. So, extra care should be taken while melting. Sintering in commonly conducted in party ovens and Tab. ovens [10, 11]. These sintering systems are called conventional sintering. The compacted metal powder needs to be sintered in order to gain mechanical strength. There is also some porosity present in compacted specimens. This can be reduced by sintering since the titanium in the alloy is highly reactive to the atmospheric gases such as oxygen, nitrogen, etc. the tubular furnace with inert gas supply is chosen. The compacted alloy is sintered at 1320 °C for 45 min. The sintered workpiece is cooled to room temperature and then the secondary finishing operations are done. In our case, no secondary operation was performed. Sintering is usually done at 2/3 rds of the melting point. Assuming the Melting point of this alloy to be around 1350 °C, the right sintering temperature works out to 900 °C. Most literature on sintering of Iron-Titanium alloys gives sintering temperatures of between 950 and 1200 °C. Literature is available only for Titanium alloys mixed with Iron powder and not Iron mixed with Titanium. Hence, our work is a new work and for this combination, sintering data are not available. Here, the sintering temperature chosen is very high at 1320 °C. Hence, some partial melting is to be expected, which has happened as seen in Fig. 28.3. The dendrites seen in Fig. 28.3 confirm partial melting.

Normally, Powder metallurgy gives the final finished product. But, Secondary finishing may be performed in rare cases. This may just consist of some fine surface finishing. A sintered product can be finished in a way similar to cast products. Normally, finishing is done to improve corrosion resistance, mechanical properties, wear resistance, porosity and surface finish. Various finishing operations include–plating, coating, deburring, brazing, welding furnace treatment, etc. Oil or resin impregnation is also used in some cases, especially in self-lubricating bearings. Oil to the extent 20–30% is absorbed in this case. Resin impregnation is also used to improve machinability. Coining or sizing is sometimes done to modify surface properties. This process also provides stricter dimensional control.

28.2.5 Testing of the Alloy

Testing consists of mechanical tests like hardness, tensile and impact tests. Here only hardness and impact tests have been carried out. After this, the material is normally characterized using XRD, SEM, Microstructure analysis, SEM-EDS, etc. and the mechanical properties are correlated to the structure. In our case, XRD and microstructural analysis were done.

X-Ray Diffraction

X-Ray diffraction (XRD) is one of the simplest characterization techniques, which can be used to give information about the presence of phases or perform elemental analysis. Electron Dispersive Spectra is also sometimes used as an attachment to XRD.

XRD for both the alloys was carried out. Hardness was taken for four locations and the average was computed. Impact test was done on both the samples and microstructures of the samples were taken with the help of metallurgical microscope.

28.3 Results and Discussion

28.3.1 XRD Results

XRD shows one major peak in both samples. As seen in the XRD peaks, and one peak in the first sample and two peaks in the second sample. The hardness as seen from Table 28.1 is also more in the second sample. So, we can infer from these two data that there is a possibility of some intermetallic formation in both samples, with higher volume of intermetallic in the second sample. The XRD peak in the second sample is also strained, with the horizontal striated part being broader. This again is an indication that there may be more of intermetallics in the second sample. Intermetallics being brittle, in all probability also give rise to strain, which is reflected in the XRD pattern. Higher Iron content in the second sample could have led to intermetallic formation (Fig. 28.2).

The peak in the second sample is slightly lower in intensity. Combined with the hardness results, which indicate the presence of intermetallic, it is possible that a Ti-Zn intermetallic has formed. But a very high sintering temperature chosen could have softened the hardening effect of the intermetallic and so hardness values are lower than that reported in literature. Literature has reported intermetallics of hardness 250 HV as has been already mentioned. $TiZn_{16}$ and Ti_3Zn_{22} are two of the intermetallics mentioned in literature.

Table 28.1 Shows hardness comparison for 88Fe-10Ti-2Zn and 93Fe-5Ti-2Zn

Sample	Hardness HV 0.5				Average hardness HV
Sample 1 88Fe-10Ti-2Zn	131.23	129.56	130.66	132.66	131.027
Sample 2 93Fe-5Ti-2Zn	139.66	141.05	138.02	140.05	139.57

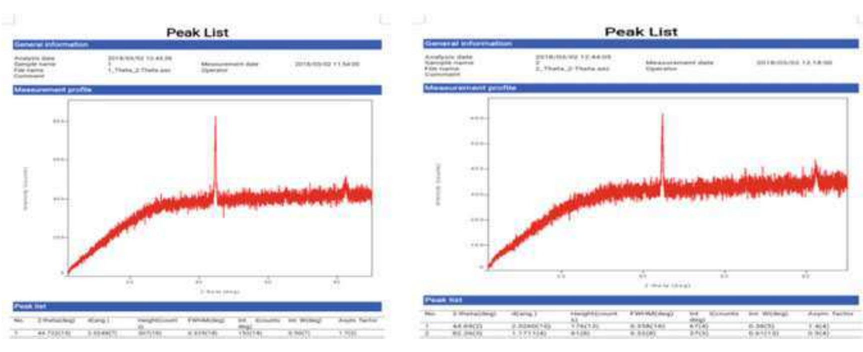

Fig. 28.2 XRD for: 93Fe-5Ti-2Zn sample and for 88Fe-10Ti-2Zn samples, respectively

28.3.2 Microstructure

Normally, powder metallurgy structures show spherical or nodular morphology of structure, especially when seen under the Scanning Electron Microscope. Figure 28.3 shows a dendritic structure. Generally, components manufactured by powder metallurgy do not exhibit dendrites. But, the presence of dendrites, in this case, can be attributed to some partial melting and cooling.

So, it is possible that at the high temperature of 1325 °C, some partial melting of lower melting elements has occurred. In Sample 1, this partial melting could have softened or dissolved some intermetallics. However, Fig. 28.3 does not show any dendrites and so it can be assumed that partial melting has not occurred, with the result that the Sample 2 is harder Zinc has a melting point of 420 °C and since Zinc is present in this powder mixture, we can say with fair degree of certainty that Zinc powder has melted and solidified. It is also seen from the microstructures that porosity is less.

28.3.3 Hardness Test Results

Hardness tests are the simplest tests, which give wealth of relevant information. They are generally used to give an idea of precipitates or intermetallics. Hardness tests should be used in conjunction with XRD or SEM in order to give meaningful results and come to accurate conclusions.

Literature reports a hardness of around 250 HV for conventionally sintered Iron powder. There is a lot of work available on Ti-Al intermetallic sand and Ti-Cu intermetallics. Ti-Cu intermetallics have hardness of the order of 630 HV. This could be due to the presence of nanoparticles. When the compaction pressure is very high, nanoparticles have been reported in literature. It is a known fact that nanoparticles show unusual properties like very high hardness. In our work, since hardness is on the lower side, there is very little possibility of nanoparticle formation. There is very little

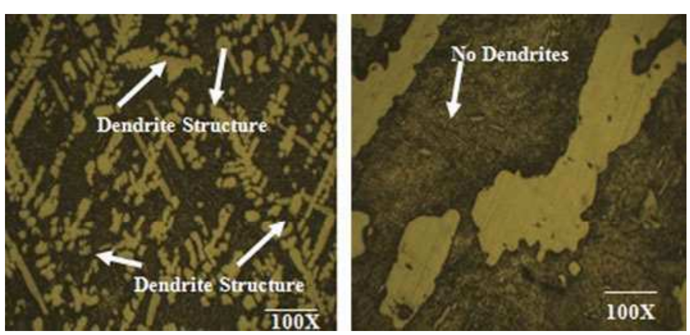

Fig. 28.3 Microstructures for samples 1 and 2, respectively

published work on Ti-Zn intermetallics, especially pertaining to hardness. Studies pertaining to phase stability are reported in literature [6]. In our work, hardness is on the lower side. The alloy has been sintered at 1320 °C. Normally, sintering temperature should be above 2/3 rd of the melting point of the constituent elements. The melting point of Iron is 1500 °C So, choice of sintering temperature is correct, but sintering time is only 45 min. It is possible that a higher sintering time could improve hardness values. Literature shows the hardness value of Titanium increases up to 0.13 wt% of Iron [12]. But, generally, Titanium being softer than Iron, we can expect Fe-Ti alloys to be less harder than Pure Iron. Better control over the sintering time and temperature could also yield better hardness values.

28.3.4 Impact Test Results

Impact value for Sample 2 (93Fe-5Ti-2Zn) was 66 J and for Sample 1 was 73 J (88Fe-10Ti-2Zn). Impact values are a combination of strength and ductility. Earlier it was enough to have good strength or good ductility but in recent times a good combination of both the properties is very much essential for a material to be accepted by the customer. A good impact value indicates a good combination of strength and ductility. However, from the microstructure analysis, Sample 1 shows some partial melting and solidification (as evidenced by the dendrites) and hence, ductility could be more in Sample 1. So, even though hardness is slightly low, it is made up by the higher ductility and as Impact strength the overall effect is higher impact strength is evident in Sample 1. Sample 2, though having higher hardness could possibly be exhibiting very low ductility due to the presence of intermetallics and also absence of any melting. XRD data also confirm strain hardening due to intermetallics. This could be the reason for brittleness in Sample 2. So, even though strength may be high, the bad impact of low ductility could have resulted in lower impact strength in Sample 2.

28.4 Conclusions

Hardness value for the second alloy 93Fe-5Ti-2Zn is higher showing that probably intermetallics are more in this alloy. However, the overall hardness in both alloys is on the lower side. This may be due to the high sintering temperature used and possibility of some partial melting. Impact strength of the first alloy 88Fe-10Ti-2Zn is slightly higher possibly due to lower amount of intermetallics in this sample. The microstructure of 88Fe-10Ti-2Zn shows some partial melting and presence of dendrites, which are not seen in Sample 2, 93Fe-5Ti-2Zn.

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