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ALUMINIUM AND IRON METAL MATRIX COMPOSITE REINFORCED WITH INTERMETALLIC COMPOUNDS AND SECONDARY PHASE PARTICLES

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ABSTRACT

The objective of the present work is to investigate the formation of intermetallic compounds such as iron alumindes and oxides during the formation of an alloy by melting commercially pure aluminium (Al) billets and adding iron powder (Fe) at 750 ºC in the molten aluminium. The exothermic reaction during the addition of 'Fe' in the molten 'Al' resulted in the formation of more amounts of intermetallic compounds because of rise in temperature. The composition of the 'Al' and 'Fe' powder were varied to obtain various alloys. The optical microscopy examination revealed the presence of various intermetallic compounds and oxides in the matrix. The Rockwell hardness survey performed on various samples exhibited a maximum hardness value of 79 in 'B' scale for the alloy composition of 60% Al and 40% Fe. A maximum shear strength of 288Mpa was obtained for the samples containing 65% Al and 35% Fe.

Keywords: *Intermetallics, Ironaluminide and Microstructure*.

1. Introduction

Aluminium is a chemical element in the boron group with symbol 'Al' and atomic number 13. It is silvery white, and it is not soluble in water under normal circumstances. Pure aluminium possesses low strength and toughness and its strength can be increased with the addition of various elements. Aluminium is alloyed with elements like 'Cu', 'Si', 'Mg', 'Zn', 'Fe' and other elements. The solubility of aluminium in iron at 400 ºC is 11.5% which is shown in Figure 1. Addition of excess 'Al' in 'Fe' results in the formation of intermetallic compounds such as FeAl, Fe_3Al, $FeAl_2$ and $FeAl_3$. Presence of intermetallic compounds and oxides reinforces the softer matrix aluminum and increases the mechanical properties. These intermetallic compounds are formed as a result of reaction between two or more metals which are mixed together in certain proportions. These intermetallic compounds exhibit neither metallic nor ceramic properties. Iron aluminides are compounds formed between 'Fe' and 'Al'. The phase diagram drawn between Al-Fe reveals the formation of intermetallic compounds like Fe_3Al, FeAl, $FeAl_2$, Fe_2Al_5 and $FeAl_3$. The properties of iron aluminide is dependent on number of aluminium atoms present in the compound. The presence of more aluminium atoms in the intermetallic compounds such as $FeAl_2$, Fe_2Al_5 and $FeAl_3$ is detrimental to strength properties and induces brittleness. On the other hand, Fe_3Al and FeAl, which have a high iron composition, are used as structural materials because of their good wear resistance, oxidation resistance, corrosion resistance and specific strength properties.

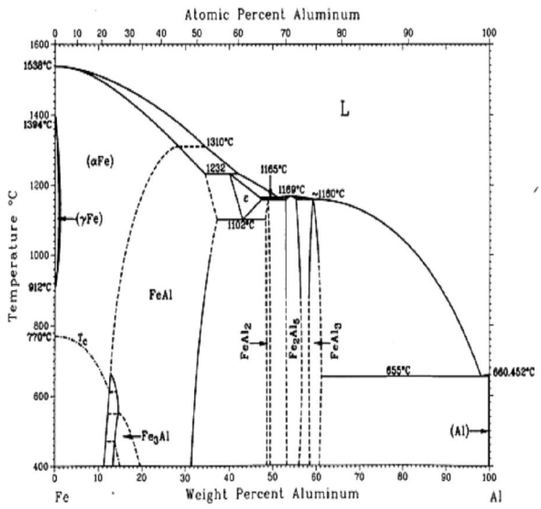

Fig. 1. Fe-Al Phase diagram (ASM Hand Book)

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In pure elements and solid solutions, the atoms are bound together with metallic bonds. In the case of intermetallic compounds covalent bond is formed which alters the crystal structure, chemical, mechanical and electrical properties. Intermetallic compounds such as Fe3Al and FeAl are among a variety of different intermetallic compounds that can be used in heterogeneous metal (Al)-intermetallic materials and intermetallics that can be used for corrosion resistance applications at higher temperature This alloy system containing Al-intermetallic compounds having nearly homogeneous composition, exhibits properties varying from intermetallics to aluminium alloys. The literature revealed that the formation of Fe-Al intermetallic compounds took place during melting of these elements and during powder compacting and sintering of these powders.
Wangyu Hu et al [1] revealed the formation of nanocrystalline iron aluminide intermetallic compounds by mechanical alloying, sintering and high velocity oxy-fuel (HVOF) thermal spraying from the elemental powders. The strength, ductility and toughness of the samples were increased by maintaining grain size to the nanocrystalline level. Jiqiang et al [2] produced an ultra fine grained Fe-40Al intermetallic compound. The X-ray diffraction of the Fe-40Al intermetallic compound revealed the presence of FeAl phase. The Fe-40Al intermetallic compound presented ultra fine grains size in the range of 100-600 nm because of high nucleation rate and the low growth rate.

The fabrication process of the metal-intermetallic materials is one of the most important areas of research. Several research works presented the capabilities of formation of intermetallic compounds, metal-intermetallic compound and metal-ceramic combination. These include solidification processing, powder metallurgy (PM), coating technology and layered manufacturing. Among these methods, PM is the most commonly used because of its ability to produce a wide range of compositions, its phase and property control as well as shape-forming capability. In the present research work, the Al-FeAl intermetallic compounds fabrication route consist of melting the solid aluminium billets and keeping 'Fe' powder in solid state. In the first method 'Al' solid billets and 'Fe' powder are mixed together and then heated to 750 ºC. At this temperature the molten aluminium reacted with 'Fe' powder to form Al-FeAl rich phase. This alloy is then heat-treated for 10 hr in a muffle furnace. In the second method 'Fe' powder is added with the molten aluminium followed by annealing the alloy for 10 hr at 600 ºC to form Al-FeAl system. During the solidification process oxides such as Al_2O_3 and FeO_2 are formed which may have strengthened the Al-FeAl alloy.

Prichard Paul [3] conducted experiments by adding silicon to Fe-15%A1 alloy which produced faceting on powder particle surfaces during vacuum sintering above 1200°C. The faceting planes occurred on the low index planes with high symmetry. The faceting was due to a change in the solid-vapor surface energy as a function of crystallographic orientation. The extrusion of Fe-Al-Si alloys produced a wire texture, which nearly doubled the yield strength of the material in the extrusion direction compared to the transverse direction. Although the extruded alloy was fully dense, the tensile properties in the extruded direction do not represent the mechanical properties of a sintered microstructure with randomly oriented grains. The yield strength of sintered powders was comparable to the extruded powders in the same heat treatment condition. The yield strength and fracture energy of the fine grain material with approximately 10% porosity exceeded the yield strength and fracture energy of coarse grain material with approximately 3% porosity. Therefore, the processing parameters are broad enough to develop desirable mechanical properties with a wide range of microstructures. The next phase of development would be to incorporate these processing parameters into an MIM process to address the interaction of the powder with binder systems. The uniform porosity and fine grain size of the sintered Fe-15A1-2.8Si alloy, which was heat treated at 6oo ºC, developed the best combination of strength and toughness. This indicates the silicon atoms are more beneficial in a solid solution rather than in an ordered precipitate.

The objective of this work is to produce Al-Fe alloys possessing optimum mechanical properties. Addition of 'Fe' in 'Al' to very high level decreases strength because more amounts of brittle phase is formed in the alloy. Therefore, an optimum level of 'Fe' percentage should be fixed for obtaining maximum strength. In the present work 'Fe' content is varied from 20-40 % and 'Al' content 60-80 % to produce various alloys. In the molten aluminium 'Fe' powder is added which produces an exothermic reaction. This reaction generates heat and this heat partially melts the 'Fe' powder to form intermetallic compounds with 'Al'.

2. Experimental Procedure

2.1 Material

The iron powder 4-8 μm particle size having purity 99.9% is used in this study. Wrought Aluminium having 99.5 % purity with 0.2%Si, 0.1%

Fe, 0.04% Mg, 0.1%Mn and 0.05%O is used. The purchased rod is of 20 mm in diameter is cut to billets of 20mm in length.

2.2 Experimental setup

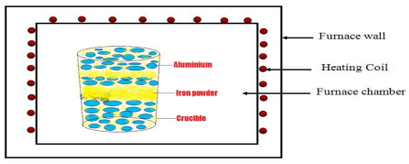

Fig. 2. Experimental setup

The muffle furnace hgaving a heating chamber of 380 mm length and 180 mm width is used. The heating rate of this furnace can be varied to required level. The maximum temperature of the furnace is 1000 ºC. Small crucibles made with clay are used for melting the aluminium metal. The experimental setup is shown in Fig. 2.

2.3 Experimental procedure

The billets were cleaned mechanically by turning them in a lathe and finally cleaned with chemicals like HCl and NaOH. The 'Al' billets were weighed and placed inside the crucibles and heated inside the furnace. The crucibles were heated upto 750 ºC and kept inside the furnace for 1 hour. In the molten 'Al', 'Fe' powder is added to initiate exothermic reaction. Because of this exothermic reaction the temperature inside the crucible rises to 1600 ºC. Further, this alloy is kept at 750 ºC for two hours and given a annealing heat-treatment for 16 hours at 550 ºC.

Table 1: Annealing Temperature for Various Composition

S.No	Composition in wt. percentage	Melting temperature	Annealing temperature 600°C and time in h
1	80 % Al and 20% Fe	750 °C and soaking at this temperature for 2h	19
2	80 % Al and 20% Fe		16
3	75 % Al and 25% Fe	750 °C and soaking at this temperature for 2h and mechanically agitated	16
4	65 % Al and 35% Fe		16
5	70 % Al and 30% Fe		16
6	60 % Al and 40% Fe		16

The specimens obtained from the furnace were machined in a lathe to obtain circular discs as shown in Figure 3 (a) and (b). Square and circular specimens of 6 x 6 mm and 4 mm in diameter (for metallography and shear test) were cut using wire-cut electric discharge machining technique. The WEDM specimens are shown in Figure 3 (c)

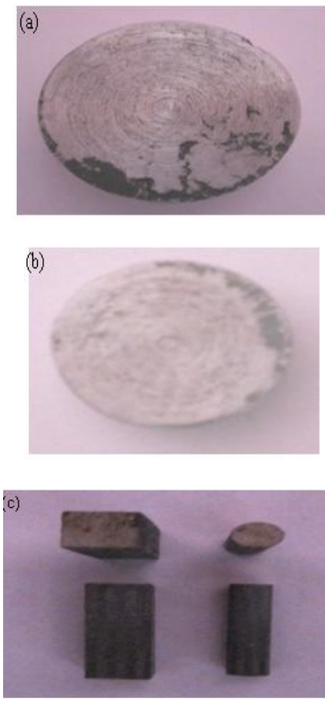

Fig. 3 (a) and (b) Shows Al-Fe Partial Melt Turned Samples and (c) shows Wire-cut EDM Samples.

The metallographic samples were polished using various grades (upto 1200 grade) of silicon carbide paper to obtain mirror finish. These samples were finished with Al_2O_3 slurry to obtain mirror finish without any scratch marks. The polished specimens were etched with a chemical mixture containing 5 ml HCl + 10 ml HNO_3+3 ml HF+ 50ml H_2O for 1minute. The specimens were rinsed in water and dried using a drier.

3. Result and Discusion

3.1 Optical microscopy

The optical micrographs shown in Figure 4 (a)-c) correspond to the sample1 processed at 750 ºC and annealed at 550 ºC for 19 hour inside the furnace. The Figure 4 (a) shows three different regions marked as

region ‘A’, region ‘B’, and region ‘C’. These three regions are rich with intermetallic compounds such as FeAl2, Fe2Al5 and FeAl3 and oxides like Al2O3 and FeO2. Further, away from these dark regions a yellowish region with fine grains of ‘Al’ is noted. This region is rich with pure aluminium. Adjacent to this region on both sides fine intermetallic compounds which impart dispersion strengthening effect to this soft region is seen. The micrograph shown in Figure 4 (b) reveals three distinct regions containing recrystallized regions rich with alternate grains of aluminium and intermetallic compounds. The grain boundaries are precipitated with ‘Fe’. The recrystallization is because of pinning of these ‘Fe’ atoms along the grain boundaries and migration of ‘Fe’ atoms into ‘Al’ matrix and subsequent formation of newer grain boundaries. In Figure 4 (c) a dark region which resembles like a large spear at the centre of the micrograph with serrations at the sides is observed. Which indicates the formation of interemtallic compounds and its growth into the ‘Al’ grains because of long annealing time and diffusion of ‘Fe’ atoms into the ‘Al’ matrix.

The micrographs shown in Figure 4 (d)-(f), correspond to the sample 2 as given in Table 1. This sample is rich with 20% ‘Fe’ and annealed for 16 hours at 550 ºC is mechanically agitated to obtain homogeneous alloy. Because of this the micrographs shown in Figure 4 (d)-(f) reveals the presence of fragmented intermetallicc compounds such as $FeAl_2$, Fe_2Al_5 and $FeAl_3$. In Figure 4 (d) the thick grain boundaries are visible and adjacent to the grain boundaries finger print like intermetallic compound formation is noted. The circle marks in Figure 4 (d) shows the presence of intermetallic compounds at various regions and their distribution is even. The Figure 4 (e) shows a net work of thick grain boundaries and a combination of fine and coarse ‘Al’ grains. The grain boundaries are rich with ‘Fe’ because segregation of ‘Fe’ along the grain boundaries is due to diffusion towards higher defect zones. The region within a circle marked as ‘A’ has very fine grains rich with ‘Fe’. On the other hand regions within circle markings ‘B’, ‘C’ and ‘D’ consists of fine grains with low ‘Fe’ atoms. The spherical and mesh grain boundaries are noted in the published work by Chakraborthy et al [4]. In Figure 4 (f) large and fine grains of aluminium are seen. Adjacent to these grains a dark region running across the micrograph is seen which is rich with intermetallic compounds such as $FeAl_2$, Fe_2Al_5 and $FeAl_3$.

The optical micrographs shown in Figure 5 (a)-c) correspond to the sample 3 processed at 750 ºC and annealed at 550 ºC for 16 hour inside the furnace. The Figure 5 (a) shows three different regions marked as region A and region B. These three regions are rich with intermetallic compounds such as $FeAl_2$, Fe_2Al_5 and $FeAl_3$ and oxides like Al_2O_3 and FeO_2. Along the grain boundaries newly formed grains are formed because of diffusion of ‘Fe’ and subsequent recrystallization and grain boundary formation. Further, away from these dark regions a yellowish region with fine grains of ‘Al’ is noted. This region is rich with pure aluminium. Adjacent to this region on both sides fine intermetallic compounds which impart dispersion strengthening effect to this soft ‘Al’ region is seen. The micrograph shown in Figure 5 (b) reveals two distinct regions containing recrystallized regions rich with alternate grains of aluminium and intermetallic compounds.

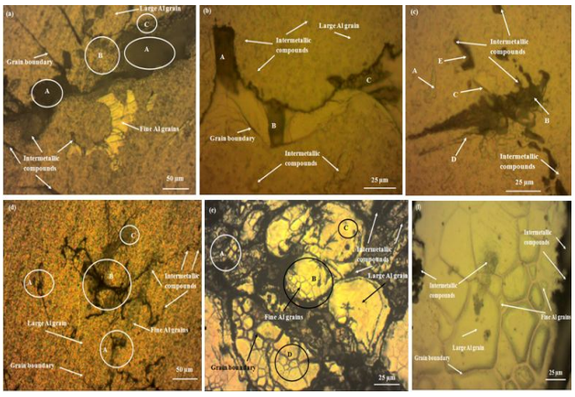

Fig. 4 (a)-(c) Optical Micrographs of Sample1and (d)-(f) Optical Micrographs of Sample 2.

The grain boundaries are precipitated with ‘Fe’. The recrystallization is because of pinning of these ‘Fe’ atoms along the grain boundaries and migration of ‘Fe’ atoms into ‘Al’ matrix and subsequent formation of newer grain boundaries. In Figure 5 (c) a dark region which resembles like a large spear at the centre of the micrograph with serrations at the sides is observed. This indicates the formation of interemtallic compounds and its growth into the ‘Al’ grains because of long annealing time and diffusion of ‘Fe’ atoms into the ‘Al’ matrix.

The micrographs shown in Figure 5 (d)-(f), correspond to the sample 4 as given in Table 1. This sample is rich with 35% ‘Fe’ and annealed for 16 hours at 550 ºC is mechanically agitated to obtain homogeneous alloy. Because of this the micrographs shown in Figure 5 (d)-(f) reveals the presence of fragmented intermetallicc compounds such as FeAl2, Fe2Al5 and FeAl3.

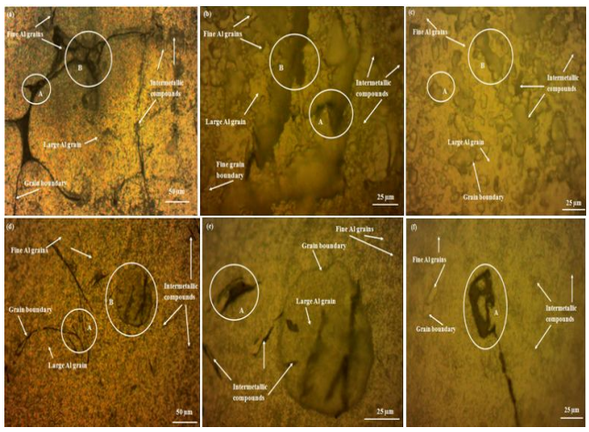

Fig. 5 (a)-(c) Optical Micrographs of Sample 3 and (d)-(f) Optical Micrographs of Sample 4.

In Figure 5 (d) the thick grain boundaries are visible and adjacent to the grain boundaries finger print like intermetallic compound formation is noted which resembles like laves phase (circle mark A). The lengthy intermetallic compounds rich with 'Fe' are observed in the published literature by Chakraborty et al [4]. The circle marks containing a sphere in Figure 5 (d) shows the presence of intermetallic compounds. The Figure 5 (e) shows a dark region contained in a circle 'A' is rich with intermetallic compounds $FeAl_2$, Fe_2Al_5 and $FeAl_3$. At few places curved interemtallic compounds are formed. The grain boundaries are rich with 'Fe' because segregation of 'Fe' along the grain boundaries is due to diffusion towards higher defect zones. The region within a circle marked as 'A' has very fine grains rich with 'Fe'. In Figure 5 (f) very fine grains of aluminium and intermetallic compounds are seen. The tail portion of this dark region runs across the micrograph. At the centre of the micrograph a dark region contained in a circle 'A' rich with intermetallic compounds is observed. The micrographs shown in Figure 5 (b) and (C) reveal the formation of long thick intermetallic compounds rich with $FeAl_3$ and $FeAl_6$ which is in accordance with the published work by Taylor [5].

The optical micrographs shown in Figure 6 (a)-c) correspond to the sample5 processed at 750 ºC and annealed at 550 ºC for 16 hour inside the furnace. The Figure 6 (a) shows two different regions marked as region 'A', and region 'B'. These three regions are rich with intermetallic compounds such as FeAl2, Fe2Al5 and FeAl3 and oxides like Al2O3 and FeO2. Further, away from these dark regions a yellowish bands run across the micrograph rich with fine grains of 'Al' is noted. This region is rich with pure aluminium. Adjacent to this region on both sides fine intermetallic compounds which impart dispersion strengthening effect to this soft 'Al' region is seen. The micrograph shown in Figure 6 (b) reveals two distinct regions containing recrystallized regions rich with alternate grains of aluminium and intermetallic compounds. A thick cylinder rich with intermetallic compounds found at the centre of the micrograph. The grain boundaries are precipitated with 'Fe' and attain light green colour because of the presence of 'Fe'. The recrystallization is because of pinning of these 'Fe' atoms along the grain boundaries and migration of 'Fe' atoms into 'Al' matrix and subsequent formation of newer grain boundaries. In Figure 6 (c) a dark region which resembles like a tree branch is seen. This type of interemtallic compounds formation indicates growth of interemtallic compound region into the 'Al' grains because of long annealing time which promotes diffusion of 'Fe' atoms and formation of intermetallic compounds into the 'Al' matrix.

The micrographs shown in Figure 6 (d)-(f), correspond to the sample 6 as given in Table 1. This sample is rich with 40% 'Fe' and annealed for 16 hours at 550 ºC is mechanically agitated to obtain homogeneous alloy. The concentration 'Fe' is more which increases the amount of interemtallic compounds present in the 'Al' alloy. Because of this the micrographs shown in Figure 6 (d)-(f) reveals the presence of more amounts of fragmented intermetallicc compounds such as $FeAl_2$, Fe_2Al_5 and $FeAl_3$. In Figure 6 (d) thick grain boundaries are visible and adjacent to the grain boundaries finger print like intermetallic compound formation is noted. The circle marks 'A' and 'B' in Figure 6 (d) shows the presence of intermetallic compounds at various regions and their distribution is even. The Figure 6 (e) shows a net work of thick grain boundaries and a combination of fine and coarse 'Al' grains. The grain boundaries are rich with 'Fe' because segregation of 'Fe' along the grain boundaries is due to diffusion towards higher defect zones. The long intermetallic compound present in the micrograph resembles the presence of laves phase. The region within a circle marked as 'A' has very fine grains rich with 'Fe'. On the other hand regions within circle marking 'B', consists of fine grains with low 'Fe' atoms. In Figure 6 (f) large and fine grains of aluminium are seen. Adjacent to these grains a dark region at the right side running across the micrograph is seen which is rich with intermetallic compounds such as $FeAl_2$, Fe_2Al_5 and $FeAl_3$. Kraishat et al [6] observed fragmented and lengthy interemtallic compounds formation in the published literature.

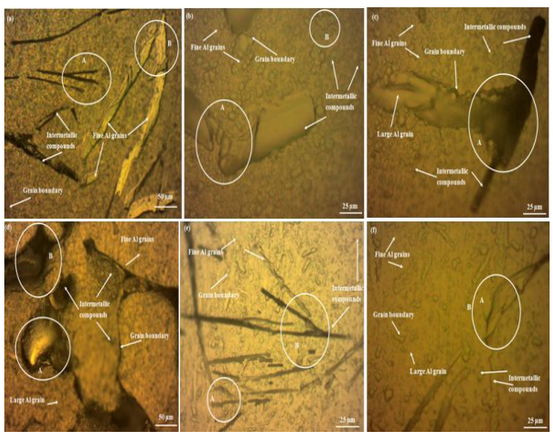

Fig. 6 (a)-(c) Optical Micrographs of Sample 5 and (d)-(f) Optical Micrographs of Sample 6.

3.2 Rockwell hardness

The Rockwell hardness values measured across the sample at twelve points were recorded. The graphical plot of the hardness values is shown in Figure 7. The hardness values were on the rise as the composition of 'Fe' was on the rise. A maximum hardness of 79 in 'B' scale was noted for the sample 6. The hardness value was in the range of 52-78 for the sample 5. A minimum value of 41 in B scale was recorded for the sample 1. This is due to less amount of 'Fe' present in the alloy.

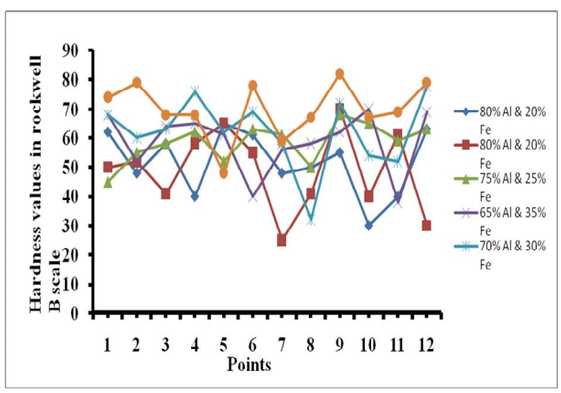

Fig. 7 The Rockwell Hardness Values of Various Samples

3.3 Shear strength

The average shear strength values are presented in Table 2. The average shear strengths of the sample 1 processed at 750 ºC and annealed for 19 hours was 278 MPa. The average shear strength of the sample 2 was only 265 MPa. The lowest shear strength was recorded for the sample 3 (Table 3) was 250 MPa. The shear strength of the sample 4 was 280 MPa. For the sample 5 the shear strength was 272 MPa. The sample 6 exhibited a shear strength of 279 MPa. The shear strength of various samples are presented as a graph in Figure 8. The variation in shear strength was attributed to variation in composition of 'Fe'and difference in annealing time and stirring of the alloy during formation of alloy.

Table 2: Shear Strength Values of Various Composition

Sl.No	Composition in wt. percentage	Shear Strength values in N/mm2
1	80 % Al,20% Fe	278
2	80 % Al,20% Fe	265
3	75 % Al, 25% Fe	250
4	65 % Al,35% Fe	280
5	70 % Al,30% Fe	272
6	60 % Al,40% Fe	279

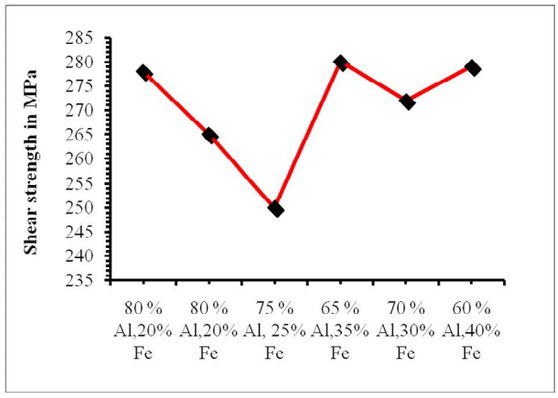

Fig. 8 The Graph Showing the Shear Strength of Various Samples.

4. Conclusions

The optical microscopy examination revealed the presence of intermetallic compounds in all the alloys. The amount of intermetallic compounds formed were on the rise for the samples containing more amounts of 'Fe'.The hardness survey carried out on the samples revealed hardness variation across the length because of presence of interemtallic compounds. A maximum hardness value of 79 in B scale was recorded for the sample 6. The concentration of Fe was at maximum level for this sample.

A maximum shear strength of 280 MPa was observed for the sample 4. The minimum shear strength was observed for the sample 3.

The formation of intermetallic compounds such as $FeAl_2$, Fe_2Al_5, $FeAl_6$ and $FeAl_3$ was observed in all the samples in varying amounts. The oxides such as FeO_2 and $Al2O_3$ was noted in few samples.

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