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ANALYSIS OF TI -NI PARTICLES ON THE MICROSTRUCTURE AND MICRO HARDNESS OF AL6082 FABRICATED BY FRICTION STIR PROCESSING

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ABSTRACT
Friction Stir Processing (FSP) has been emerging as a solid state technique to fabricate aluminium composites in recent years. In present work, FSP is applied to AL6082/ Ti-Ni in order to analyze the effect of Ti-Ni on the microstructure, homogeneity and mechanical properties. A single pass of FSP was carried out at 1120 R.P.M. rotational speed, with travel speed of 25mm/min and an axial force of 10 KN to produce composition. Alloying elements were added to three volume fractions as 5%, 6.25%, and 7%. The microstructure was studied using scanning electron microscopy (SEM). Various phases were identified by X-ray diffraction (XRD) the results shows grain size refined, micro hardness increases substantially, effect of alloying element %age on grain size is also reported in this paper.

KEYWORDS: Friction Stir Processing, Aluminium6082, Reinforced composite, Microstructure, Micro hardness

INTRODUCTION
Friction Stir Processing (FSP) is a latest material surface processing technique that can be used to refine the microstructure and in result, improved mechanical properties. The source of origin of FSP is friction stir welding (FSW) process, a solid state joining technique invented at the welding institute (TWI), United Kingdom in 1991[1,2]. Basically the FSW process joins the materials, FSP is also based on the same principle but it is used to modify the microstructure of the specific area for which the FSP is done [3]. FSP is a new material processing technique which can be used on material like aluminium, copper, magnesium etc. With the help of FSP technique a material can be alloyed up to some depth with another material without affecting the base metal up to some extent. As it is a solid state process it does not melt the metal that is being processed [4-5].

FSP is a process in which a non-consumable cylindrical tool with a concentric pin at one end of the tool is used to process the material. The rotated tool is pressed into the surface of the material and in result with a combination of frictional and adiabatic heating the material gets soften, while at the same time stiring is done by rotating tool which results in homogenous mixing of the material which further refined grain structures [6-10]. As per the research, the volume of material processed has improved properties like refine grain size etc. and increased strength. As research is going on in various field in which FSP can be utilized. The aeroplane blades, propellers of a ship can be repaired as well manufactured with a material processed by FSP technique. This technique apart from refining the surface properties also can be used to eliminate porosity in the surface [11-13]

The schematic diagram for the FSP technique is shown in figure1. In first stage the non-consumable cylindrical tool is rotated at a prefixed RPM. In second stage the tip of the rotating tool is plunged into the work piece and it generates a lot of heat by its rotation. The shape of the tool tip which is small in size fully inserted into the metal and concentric large diameter shoulder which is intended to prevent upward displacement of the material on the surface of the work piece. As the tool penetrates into the material surface, the rotating pin generates friction and adiabatic heating [14-16]. This combination of friction and heat softens the material and it helps further movement of the tool into the work piece. Due to rotation and further movement of tool it creates a stirring action and material flows around the pin. The depth of penetration is controlled by the length of pin and its penetration. Third stage shows the shoulder making contact with the surface of the work piece. The rotating shoulder expands the hot zone created on the surface of the work piece. Simultaneously it stops the upward movement of the uprooted and stirred material and results in a forging action on that material. When the tool is fully inserted into the work piece, it then travels across the metal at a specific rate; inches per minute as seen in the stage four [17-18].

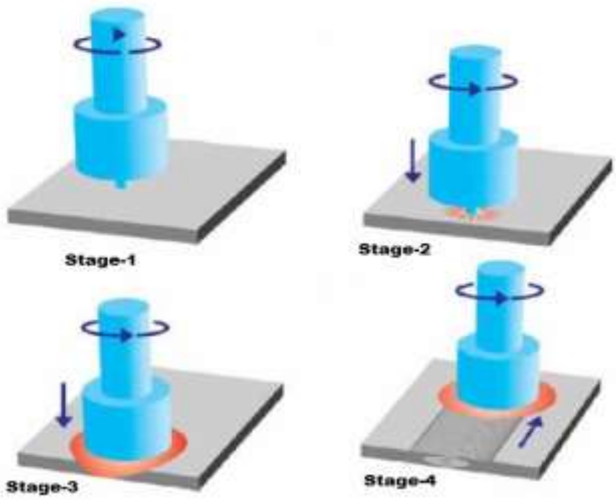

Figure 1 FSP technique

It is interesting to watch the FSP technique, as the tool progress into the metal due to heat generation within narrow region of the pin, the metal gets heated but it never melts. The peak temperature achieved in this process is around 80 to 90 percent of the melting temperature. Hence when the tool passes by, the processed work piece cools and exhibits a refined and homogenized microstructure. It means by FSP we can change the properties of the work piece within limits and the rest of the work piece got minimum influence [19-20]

EXPERIMENTAL PROCEDURE

The sheet of 12mm thickness with its chemical composition corresponds to aluminum alloy AL6082 as base alloy was used. Its chemical composition is given in table 1.

Table 1: Chemical Composition of base alloy AL6082

Element	Mg	Si	Ti	Mn	Fe	Cu	Al
Wt %age	0.40	1.3	0.1	0.49	0.28	0.16	97.27

The sheet was cut as rectangular sample of size 150mm*50mm*12mm. The SEM of as received Al6082 plate is shown in figure2. A Groove of 6mm deep was made along the centre line of the plate using wire cut electrical discharge (EDM) & compacted with Ti + Ni Powder. A pin less tool was initially employed to cover the top of the groove after filling the Ti + Ni particles to prevent the particles from scattering during FSP [28].A Tool made of M-35 HSS as shown in figure 3 was used for the present study. The chemical of tool is given in table 2

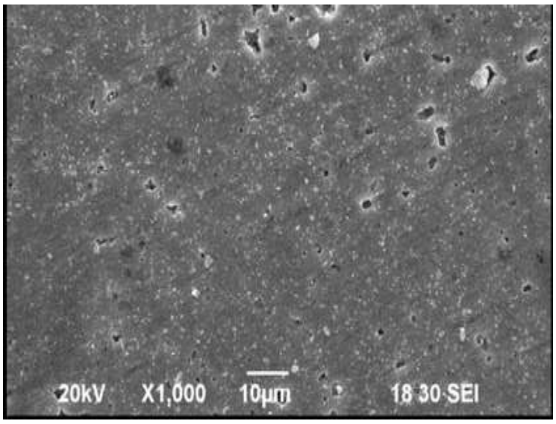

Figure 2 SEM image of AL6082 as received

Figure 3 image of tool used

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Table: 2 Chemical Composition of tool

Element	**C**	**Si**	**Cr**	**Va**	**W**	**Mo**	**Co**
Weight % age	0.8	0.33	4.0	2.0	6.0	5.0	5.0

The tool had a shoulder having diameter of 20mm, pin diameter 6.5mm & pin length 6.5mm was used. The FSP was carried out on a Vertical milling machine having 5 H.P motor with different speed & feed ratio as automatic operated. The process parameters employed were tool rotation speed 1120 R.P.M. , transverse speed 25 mm/min & the axial force of 10 KN FSP was processed using three plates of varying the width of grooves as 1.00mm, 1.25mm, 1.5mm to have the three level of volume fraction of Ti + Ni particles as 5%, 6.25%, 7.5% Specimens of 12 mm thickness were obtained by cutting the friction stir processed plate at its center perpendicular to the processing direction. All the specimens were polished as per the standard procedure. The chemical composition & SEM of the specimen was done using Scanning Electron Microscope (SEM, JEOL) in Thapar University, Patiala (India). The micro hardness was measured at CTR Ludhiana (India) using micro hardness tester at 100g applied load for 15 sec at the various locations in the specimen. The XRD of the specimen was done at Thapar University Patiala on X-ray Diffractometer (XRD, PanAlytical) to obtain the phase formed of the specimen.

RESULTS & DISCUSSIONS

Fig 4 shows the typical crown appearance of friction stir processed aluminum with Ti + Ni powder. As it can be seen from the figure that no crack, void etc. are seen on the surface of the processed specimen. The curve structure is appeared on the top surface which is similar to the conventional milling process because FSP is derived from FSW. Some trail experiments were conducted were conducted initially to set & optimize the process variable & to obtain a crack free surface because a smooth crown appearance is essential in the processed zone.

Figure 4 Crown appearance of friction stir processed specimen

Chemical composition of AL 6082 & specimen

The chemical composition of the all the three specimens named as specimen -1, specimen-2, specimen-3 having 5%, 6.25%, 7.5% of the alloying element of Ti + Ni in the base metal were compared with the base metal. The table 3 shows the comparison of chemical composition of three specimen with base metal which shows that the 5age of Ti + Ni is increased in the base metal as per the 5age of pouring powder.

Table 3: Comparison of base metal with specimen

Element		Mg	Si	Ti	Mn	Fe	Cu	Ni	Al
Wt %age	Base metal	.4	1.3	.1	.49	.28	0.16	------	97.27
	Specimen-1	0.4	1.2	0.3	0.72	0.19	0.16	0.25	96.45
	Specimen-2	0.43	1.25	0.43	0.68	0.23	0.13	0.4	96.45
	Specimen-3	0.38	1.15	0.48	0.72	0.20	0.14	0.5	96.43

Microstructure examination / measurement

Scanning electron microscope (SEM) of JEOL JSM 6510 LV at 20 KV was done at thapar university Patiala. All the FSP stir zone samples were mechanically ground & polished with 0.1 µm aluminum paste & cleaned with the

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acetone & triple distilled water. The SEM micrographs of the prepared specimen are shown in figure4. The fig 5(a) shows the grain distribution in base metal of AL6082. The distribution of the prepared specimen is shown in figure 5(b), 5(c), 5(d) with different %age of alloying elements. The distribution of Ti + Ni particles is fairly homogenous in the specimen. It is observed from the SEM test results that the Ti + Ni particles are more homogenous in specimen -1 as compared to the specimen -2 & specimen -3 which is due the large % age of pouring element in the specimen -2 & specimen-3. The average grain size of 4 – 5 µm is calculated in the specimen.

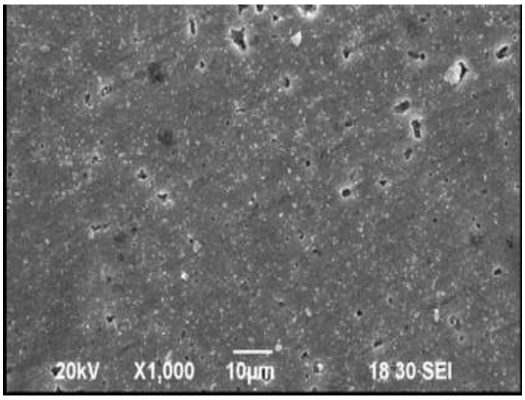

Figure 5 (a)

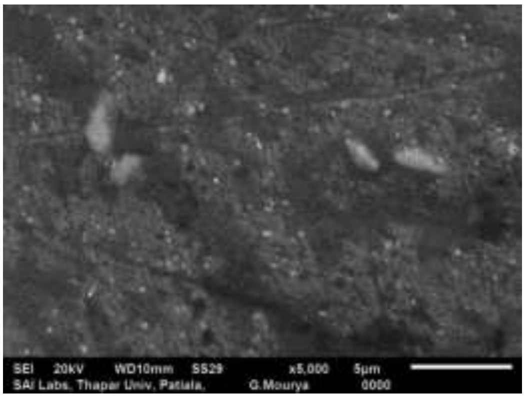

Figure 5 (b)

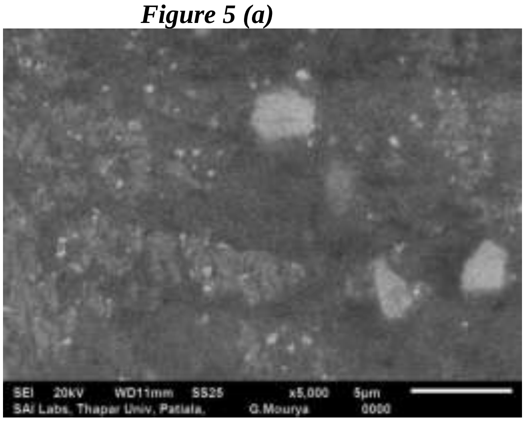

Figure 5 (c)

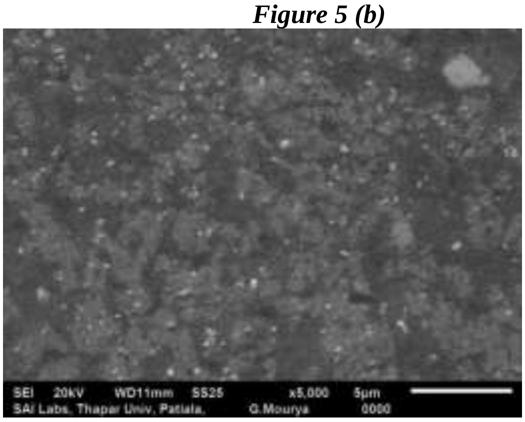

Figure 5 (d)

Phase Identification / XRD

The X-ray Diffraction test was done on all the specimen prepared by FSP is done. X-ray diffraction (XRD) is a mature x-ray technology that is widely used in the minerals industry for mineral identification and quantification. XRD uses the unique crystal structure and derived x-ray fingerprint of any crystalline material, e.g. mineral, for identification. XRD, unlike other mineral or bulk techniques such as SEM-EDS (MLA) or bulk assay (respectively), is unique in being able to unambiguously identify a mineral in a bulk or pure mineral sample, irrespective of the mineral type - sulphide, anhydrous or hydrated. While it can be used to provide gross solid solution mineral chemical changes (e.g. in carbonates, feldspars) the method is insensitive to minor or trace element changes in a mineral lattice.

Every crystalline substance is made up of an intrinsic regular arrangement of atoms in the form of its crystal lattice. When the lattice is illuminated by a collimated beam of x-rays the lattice reflects the x-rays at angles (Φ) specific to the distance spacing of the individual lattice planes (d-spacing, or “d”) and the wavelength of the illuminating x- radiation (λ)

The Bragg Equation relates these parameters:

$$n\lambda = 2d.\sin\Phi$$

The XRD of the specimen were carried at 2θ angle range from 10° - 80° with step size of 0.0130° at Thapar University, Patiala. Figure 6(a) & 6(b) shows the XRD main graphics, analyze view of specimen. Table 4(a) &

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4(b) shows the pattern list of the XRD test. In both of the main graphic analyze view the four peaks values are seen the value of this peak is given in the table 4(a) & 4(b) which shows that in specimen no1 85% of Ti + Ni+ Al phase is formed whereas in the second specimen the 26% of Ti + Ni + Al phase is formed. From the table 4(a) & 4(b) it is seen that Ni is fairly mixed with aluminum in specimen- 2 whereas in specimen -1 the Ti is fairly mixed with aluminum to generate homogeneous mixture.

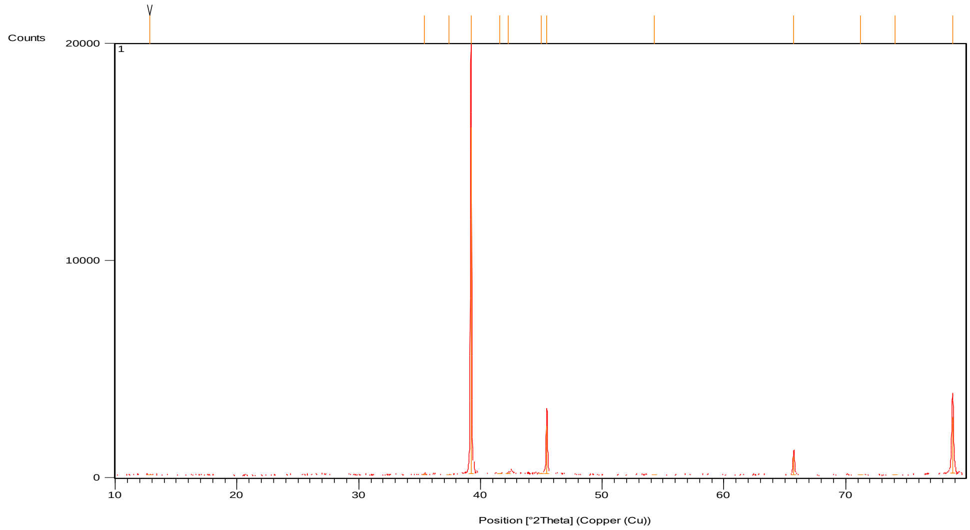

Figure 6(a) Main Graphics; Analyze View of Specimen -1

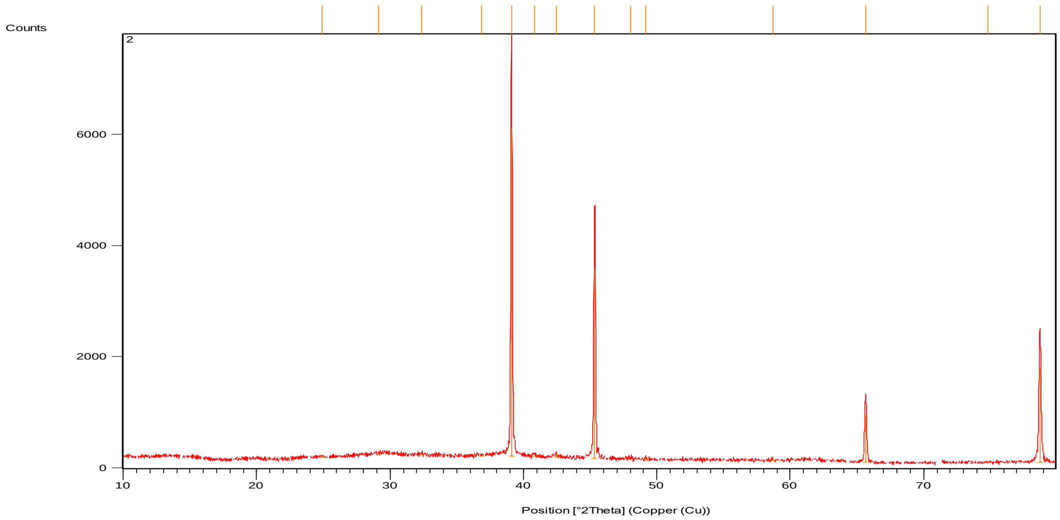

Figure 6(b) Main Graphics, Analyze View of Specimen -2

Table: 4(a) Pattern List of Specimen -1

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula	SemiQuant [%]
*	01-074-3785	47	Titanium Nickel Aluminum	-0.478	0.494	Ti (Ni0.11 Al0.89)3	85

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*	03-065-3957	26	Nickel Titanium	-0.128	0.004	Ni4 Ti3	1
*	01-072-9142	55	Aluminum Titanium	0.098	0.055	Al2 Ti	10
*	01-074-5327	29	Aluminum Nickel	0.341	0.016	Al3 Ni2	5

Table: 4(b) Pattern List of Specimen -2

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula	SemiQuant [%]
*	01-072-8274	29	Aluminum Nickel Titanium	-0.472	0.019	Ti27.55 Ni28 Al63.73	26
*	01-072-2619	14	Nickel Titanium	-0.069	0.005	Ni2 Ti	7
*	03-065-7340	41	Nickel Aluminum	0.232	0.049	Al4 Ni3	50
*	01-074-4925	11	Aluminum Titanium	-0.412	0.013	Al5 Ti2	17

Micro hardness of AL6082 & Specimen

The micro hardness of AL6082 at various volume fractions of Ti + Ni is shown in figure 7(a), 7(b) & 7(c) at various position of the specimen from the forward position of the tool. The average value of the micro hardness is 81 HV of 5% volume fraction, 110 HV of 6.25% volume faction, 120 HV of 7.5% volume faction. The microstructure changes induced by the reinforcement of Ti + Ni particles are responsible for the improvement in mechanical properties. It is also observed that from figure 7(a), 7(b) & 7(c) that the micro hardness of all the specimen goes on little bit increasing as the tool moved in the forward direction this is due to fact of the mixing of the alloy element of Ti + Ni in the base metal because of the heat flow in the forward directions.

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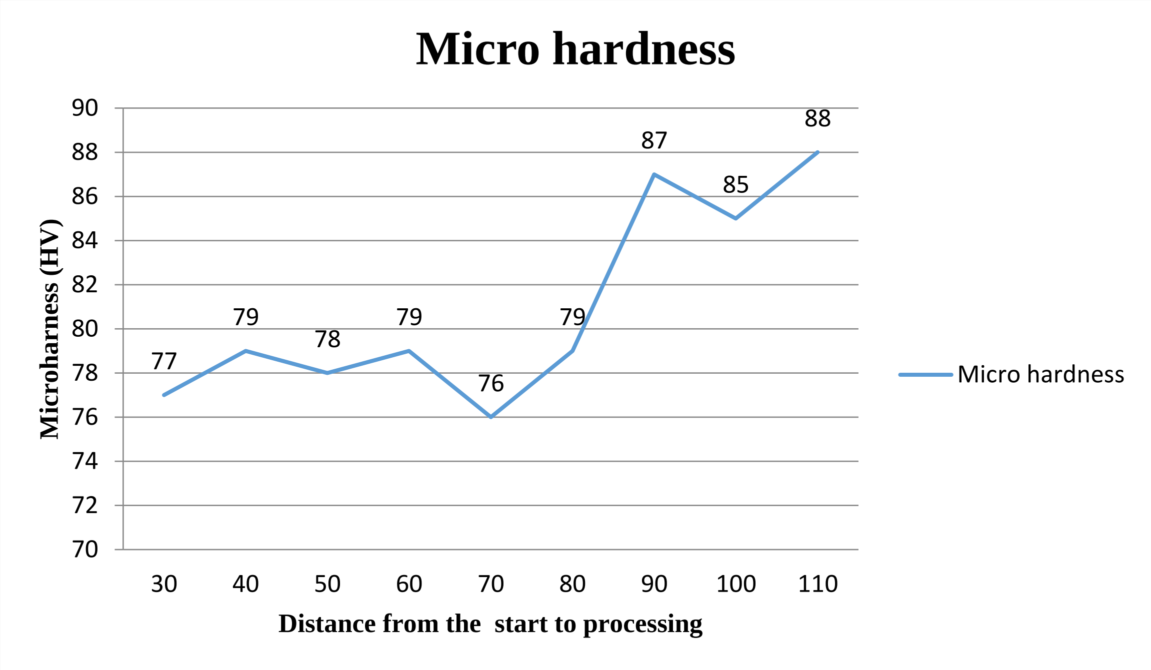

Figure 7(a) Micro hardness of Specimen-1

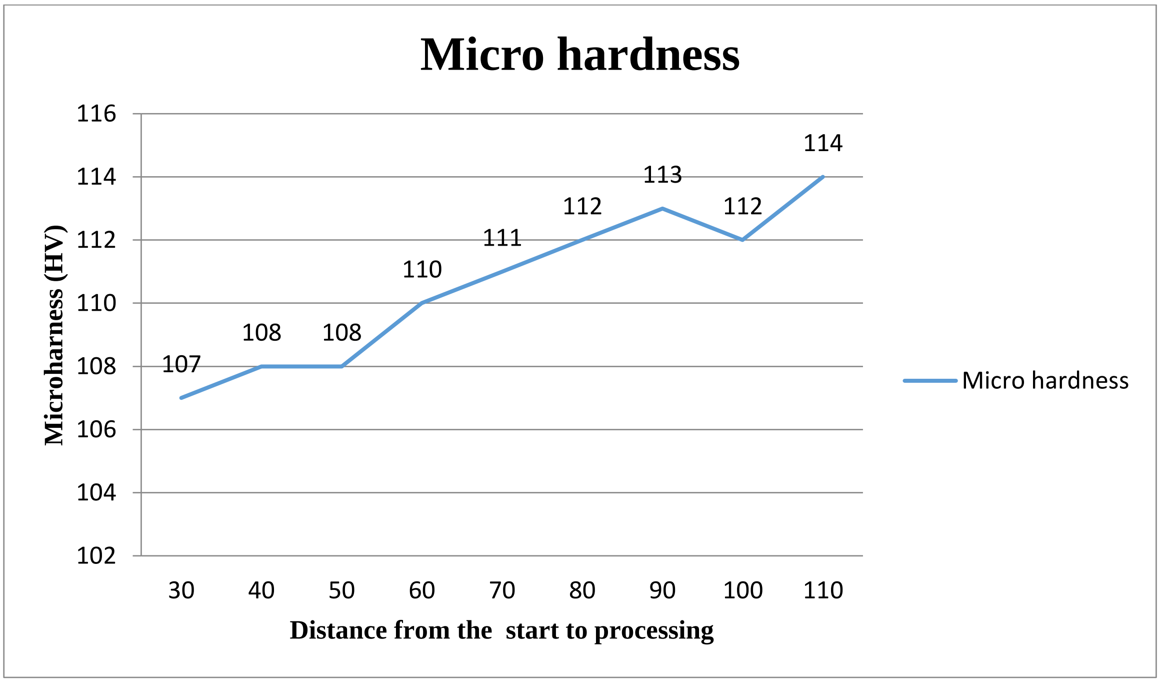

Figure 7(b) Micro hardness of Specimen-2

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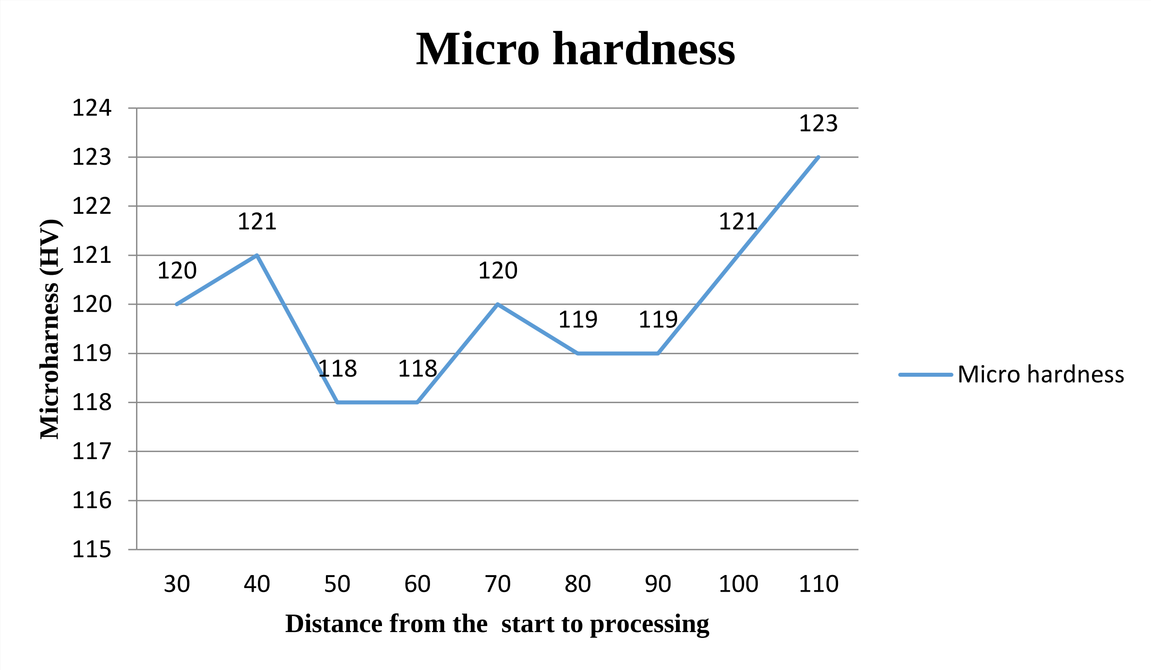

Figure 7(c) Micro hardness of Specimen-3

CONCLUSIONS

1. The average micro hardness of the specimen is increased from 81 HV to 120 HV.
2. From the EDX test the mixing of Ti is observed from .1 to 0.48.
3. From the EDX test the mixing of Ni is observed from 0 to 0.5.
4. The grain structure is more refined from the increase in %age of pouring element.

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