The effect of milling time and composition on the microhardness of Al/Al2O3 composite using design of experiments and Taguchi method

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Abstract

The effect of milling time and composition on the microhardness of Al/Al_2O_3 composite was studied. Milling time was varied from 6 to 24 hours and composition was varied from 2.5 to 10 Vol% of alumina. Microhardness data were obtained by using Vickers hardness test using 25 reading for each combination. A linear model which is based on the design of experiments was applied to determine the effect and importance of each factor. For optimizing the process a second order regression model was developed. Results shows when both the milling time and composition were increased, the microhardness increased due to the significant interaction effect between the milling time and composition. A contour plot was produced which showed that at many milling time and composition conditions at which a desired value of hardness can be achieved.

Keywords: metal matrix composites, Design of experiments, Taguchi method,

1. Introduction

There has been a wide interest in developing and producing metal matrix composite (MMC) because of their unique mechanical properties and advanced applications. The most popular type of MMC is aluminium alloy reinforced with ceramic particle [1,2]. The combination between ductility received from Aluminium and high strength from ceramic materials particles gives the Aluminium metal matrix composites (AMC`s) a wide range of applications for both the aerospace and automotive industries [1-5].

Al/SiC/ Al_2O_3 as an advanced material were produced by many researchers via mechanical alloying [3, 6-9]. Where it is a technique which has been used widely for preparation of nanostructured materials and was found as a useful method for improving the reinforcement particle distribution in both microcomposite and nanocomposite .The presence of Al_2O_3/SiC as reinforcement within the aluminium matrix affects the mechanical properties as the wear resistance and the microhardness which will be increased [10-13]

The present work can be used as an attempt to study the effect of different milling time (Hr) and volume fraction of Al_2O_3 on the microhardness of Al matrix using the design of experiments and Taguchi method.

2. Design of experiments

Experiments of this study was designed and analysed statistically based on the design of experiments (DOE) method with using the software MINITAB 15.Where the factorial experimental robust design as a powerful technique [14-17] will be used to determine the significance of the milling time and volume fraction of Al_2O_3 on the microhardness of Al nanocomposite..

To further support the design of experiment a further support by using Taguchi methods was included. Where Taguchi focuses on the importance of reduction variations.

3. Experimental procedures

As starting materials, an aluminium powder (99.5%pure; average particle size of 40 µm) and an alumina (99%pure; average particle size ~50nm) were used. A hardened steel vial with a cylindrical cavity of 60mm in depth and a 100mm in diameter and stainless steel balls with a diameter mix of 12/25 mm were used for the milling using a PM 4000 Restch planetary ball mill. The vial containing balls and 100gr of powder mixture was sealed in a glove box filled with high purity argon.

The balls to powder weight ratio was 5:1.In the nominal compositions of powder mixture the Al_2O_3 volume fraction varied from 2.5 to 10vol%.The powders were first mixed for 6 hours using a rotation speed of 100rpm, and then the powder mixtures was milled for a net time of 24 hours with a rotational speed of 4oorpm.After every 6 hours of milling, a small sample was taken out for analysis. The analysis and characterization of the samples were performed using a Vickers Microhardness tester with a load of 25gr and a loading time of 15 s.

Four levels for each factor were chosen. For milling time 6,12,18 and 24 hour of milling and Volume fraction for Alumina were 2.5,5,7.5 and 10 vol%. With each cross combination 25 readings of microhardness were taken under the same conditions which lead to a total 400 readings (4*4*25). A general full factorial design was used to determine the main effects and interaction between them

4. Results and Discussion

The factors values and levels are summarized in Table 1.For each combination of milling time and composition of alumina used as reinforcement; twenty five microhardness data were taken. The analysis of variance for microhardness readings is shown in Table 2. Linear statistical model and second order regression were used to model and analyse the experiment.

Table1: The factors and there values

Factor	Type	Levels	Values
Composition(Vol%)	Fixed	4	2.5,5.0,7.5,10.0
Milling time(Hr)	Fixed	4	6,12,18,24

Table2: Analysis of Variance for Microhardness (HV), using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Composition	3	73515.4	73515.4	24505.1	137.34	0.00
Milling time (Hr)	3	16229.2	16229.2	5409.7	30.32	0.00
Milling * Compo	9	9453.4	9453.4	1050.4	5.89	0.00
Error	384	68515.7	68515.7	178.4		
Total	399	167713.7				

4.1. Linear statistical model

Residuals are an important output of the design of the experiments which describes the error which is different between the actual hardness observations and the fitted hardness observations. Analysing these residuals can be used to verify the assumption of the following equation

$$H_{ijk}=\mu+x_i+X_j+(xX)_{ij}+\epsilon_{ijk} \quad i,j=1,2...4,k=1,2,3,....25 \quad (1)$$

Where H_{ijk} is a random variable donating the hardness,µ the overall mean effect, x_i is the effect of ith level of milling time , X_j is the effect of composition , $(xX)_{ij}$ is the effect of interaction and ϵ_{ijk} is the random error component having a normal distribution with zero mean and constant variance.

The normality assumption of Eq.1 can be tested by constructing a normal probability plot of the standardized residuals, as shown in Fig.1. Were the residuals ranked from the smallest to largest and plotted against their observed cumulative probability. The closer the data points are from the fitted line, the better they can be described by a normal distribution.

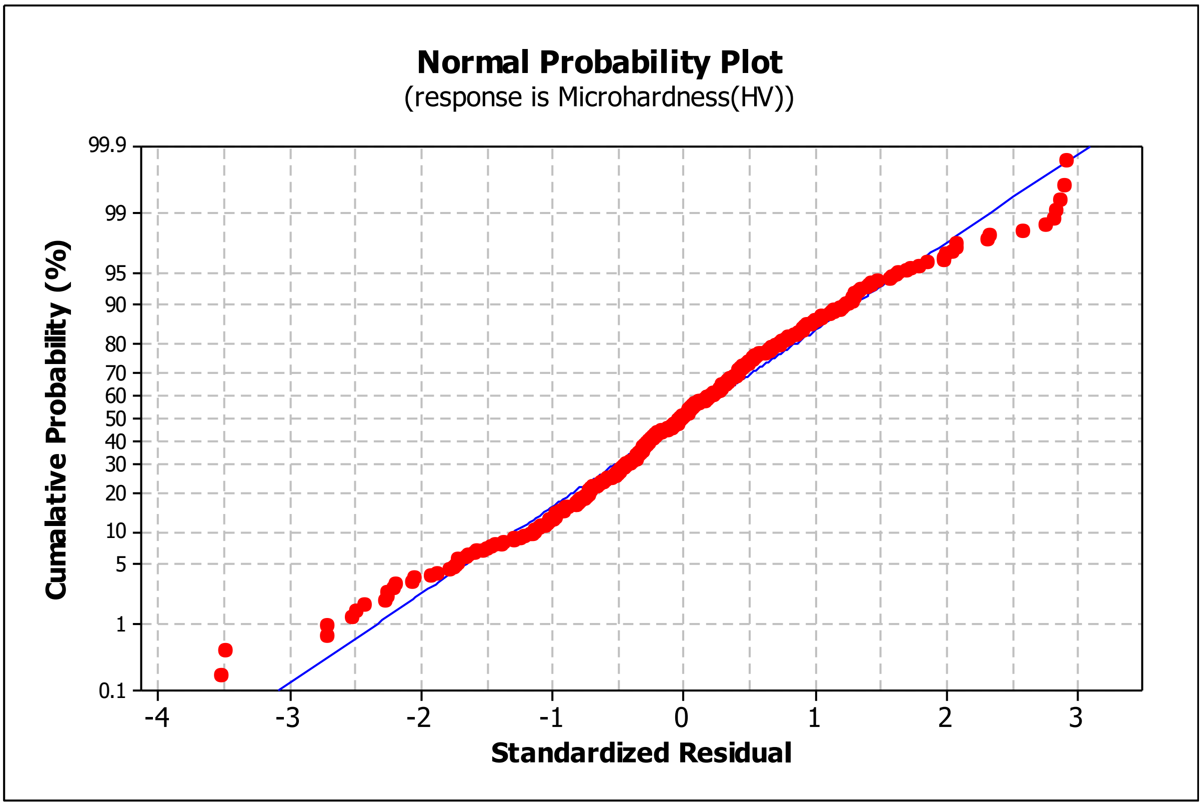

Fig.1 Normal Probability plot for the standardized residuals

Standardized residuals were plotted as a function of fitted values in Fig.2 and it shows that it's evenly distributed around the zero line. Also it doesn't show any pattern of increase or decrease. This indicates that the residual variance and the error variance are approximately constant. To check for run orders related effects, standardized residuals were plotted against the run order of the observations, shown in Fig.3.Which seems to be evenly distributed around the zero line and no random pattern patterns exist within the data points. This means that the run order isn't important factor.

After validating the assumptions of the linear statistical model, the significance of the contribution of the milling time and composition factors on the Al-Al_2O_3 composite microhardness can be examined. This was accomplished by applying the analysis of variance combined with the p-value, as shown in table 2.The main output of this analysis is the p-value, which is the smallest level of significance .Since the calculated P-value for milling time, composition and its interactions are zero, then all these values are significant at any level of significance.

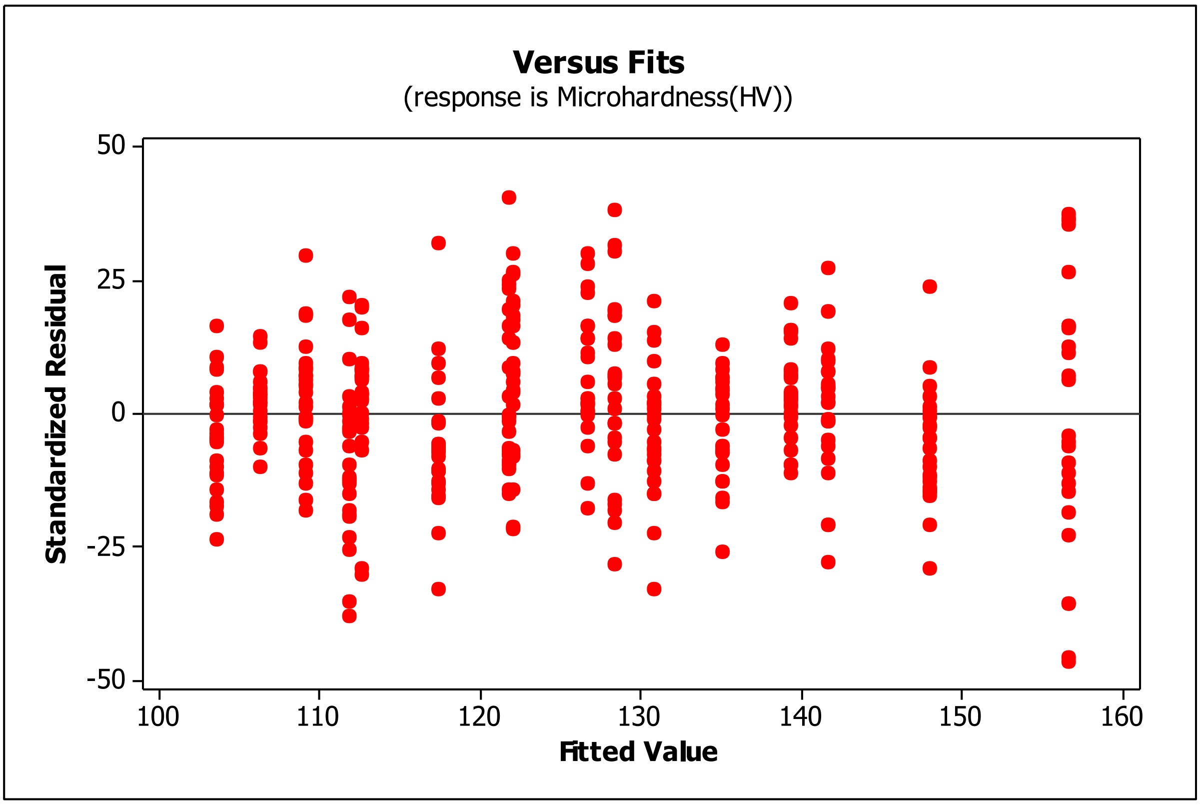

Fig.2 Fitted value vs Standardized residuals for microhardness observations

The significance of milling time and composition factors can be checked also by constructing their main effect plots, as shown in Fig.4. Where Fig.4 shows that with increasing the milling time the microhardness values increase and the same is proven for the volume fraction of the composition. From what mentioned above, it can be said, that both the milling time and composition can be considered significant factors, which agree with the analysis of variance in table 2.The interactions effect between the milling time and composition can be checked by a special interaction plots, Fig.5.

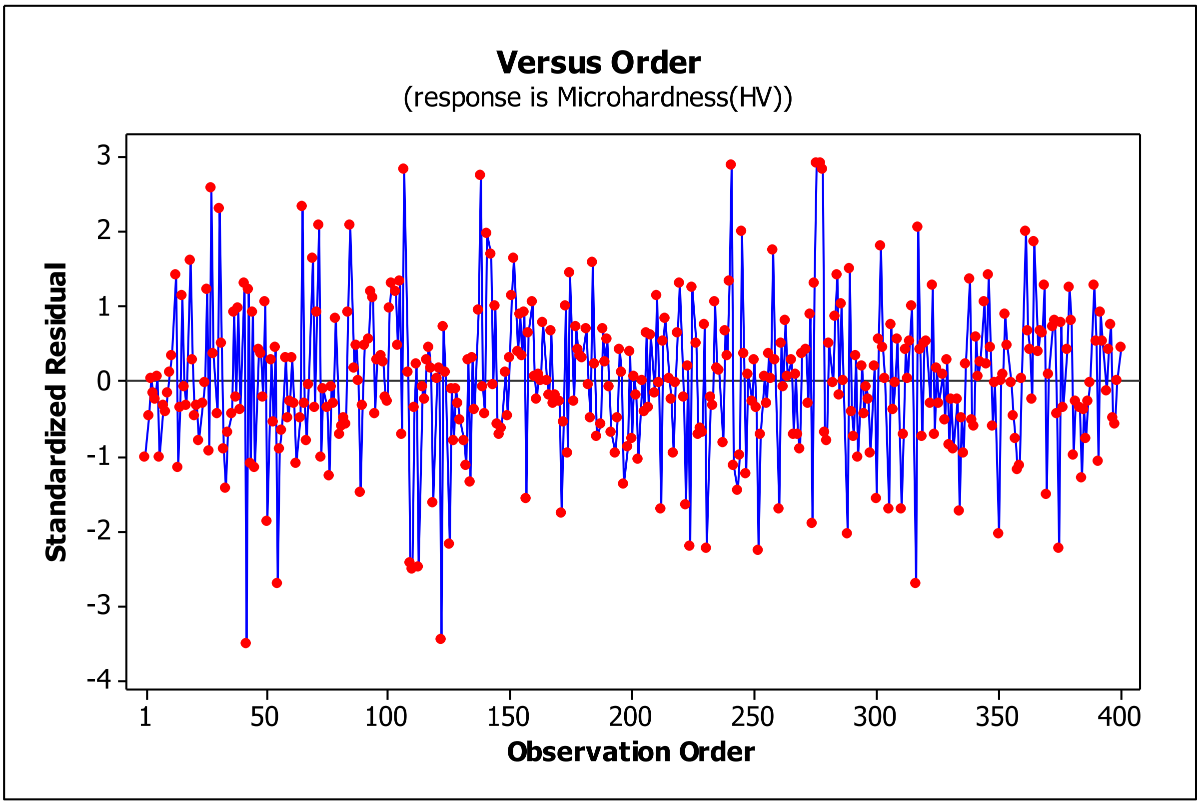

Fig3 Residuals vs. the runs order of the microhardness observations

Fig.5 shows the hardness means plotted versus the composition for each level of the milling time in one plot. These curves have approximately linear patterns, which supports the linear statistical model used. Some separation can be noticed between the different curves and that indicates that the effect of composition on the Al-Al_2O_3 composite hardness is dependent on the level of milling time. Also, the four curves are not all parallel

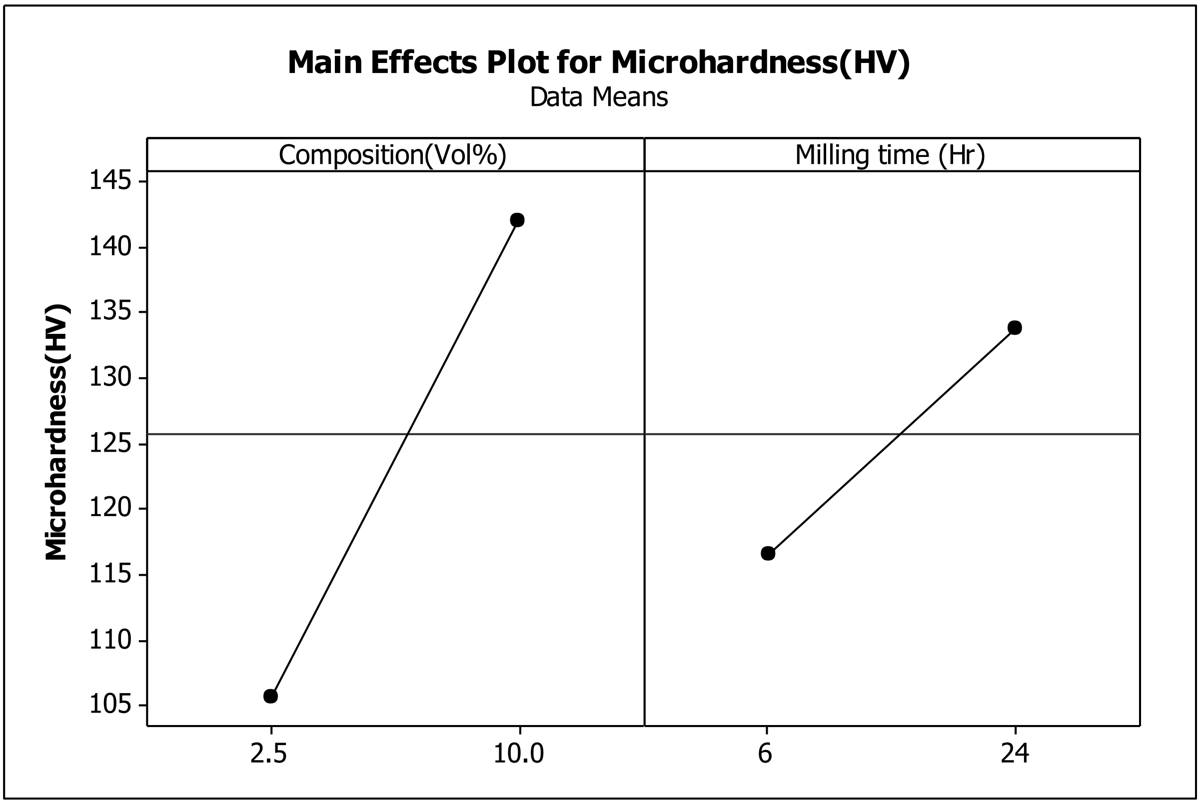

Fig.4 Main Effects plots for microhardness

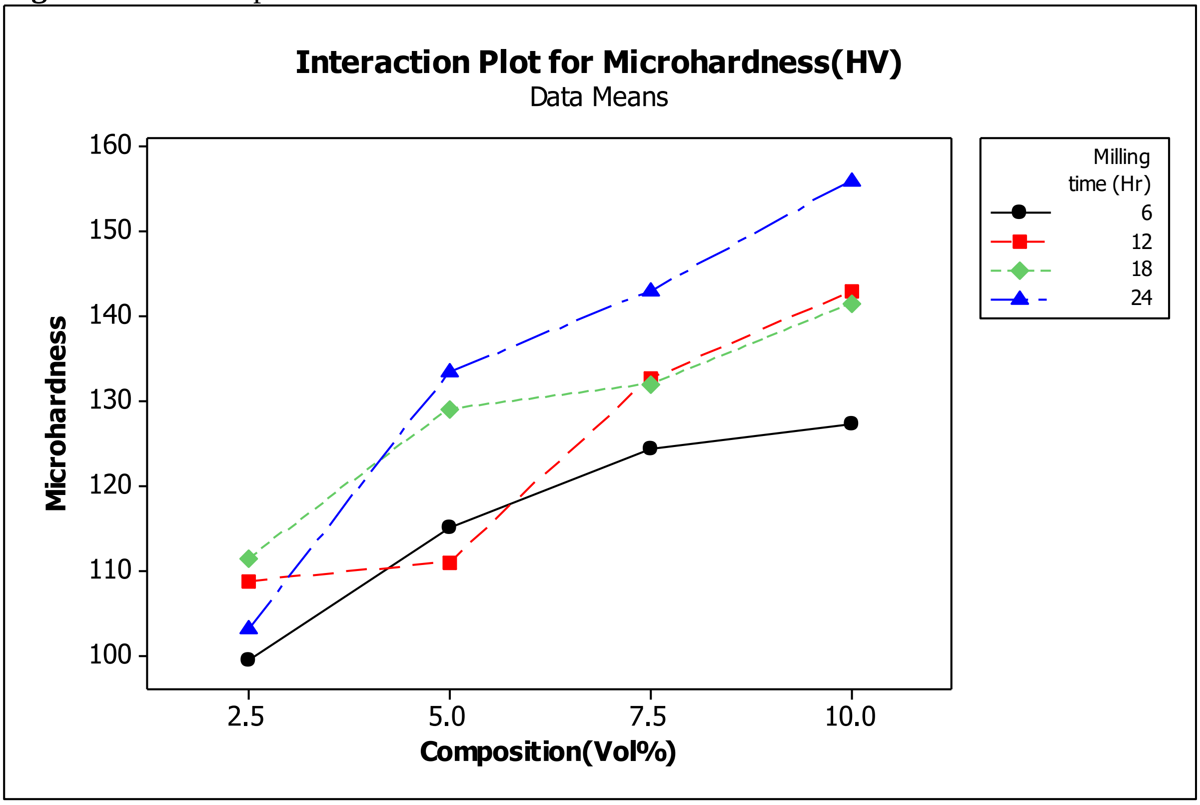

Fig.5 Interaction plot for microhardness

4.2. Taguchi Method

For further investigation of the significance of the factors by analysis of variance and the results concluded, Taguchi method was employed to support our previous talk and analysis. Where Taguchi proves itself as a strong statistical quality tool. .

Taguchi focuses on reducing variations by a loss function concept. Where the loss function recommends that the main aim of design is to produce products or processes that perform on the target with the smallest variation. By using an appropriately chosen signal to noise ratio (SN).Here in this investigation we use larger is better

$$\frac{S}{N} = -10\log\frac{1}{n}\left(\sum y^2\right) \qquad (2)$$

Where, n the number of observations, and y the observed data for each type of the characteristic, with the above S/N ratio transformation, the higher the S/N ratio the better is the result.

Taguchi recommends analyzing the S/N ratio using conceptual approach that involves graphing the effects and visually identifying the factors that appear to be significant.

Table 3 Response Table for Signal to Noise Ratios. Larger is better

level	Milling time (hr)	Composition (Vol%)
1	41.15	40.33
2	41.69	41.57
3	42.04	42.33
4	42.23	42.88
Delta	1.08	2.55
Rank	2	1

Fig.6 shows the main effects plot for SN ratios where with increasing both the milling time and composition fraction the microhardness of the Al-Al_2O_3 composites increases. From table 3 and Fig.7, it can be seen that milling time ,has the highest slope with rank equal to two, this factor affects the standard deviation highly, “mean on target “with a slight variance since it ranks equals to two for signal-noise ratio, i.e. milling time is an adjustment factor and can be used for adjusting mean on target

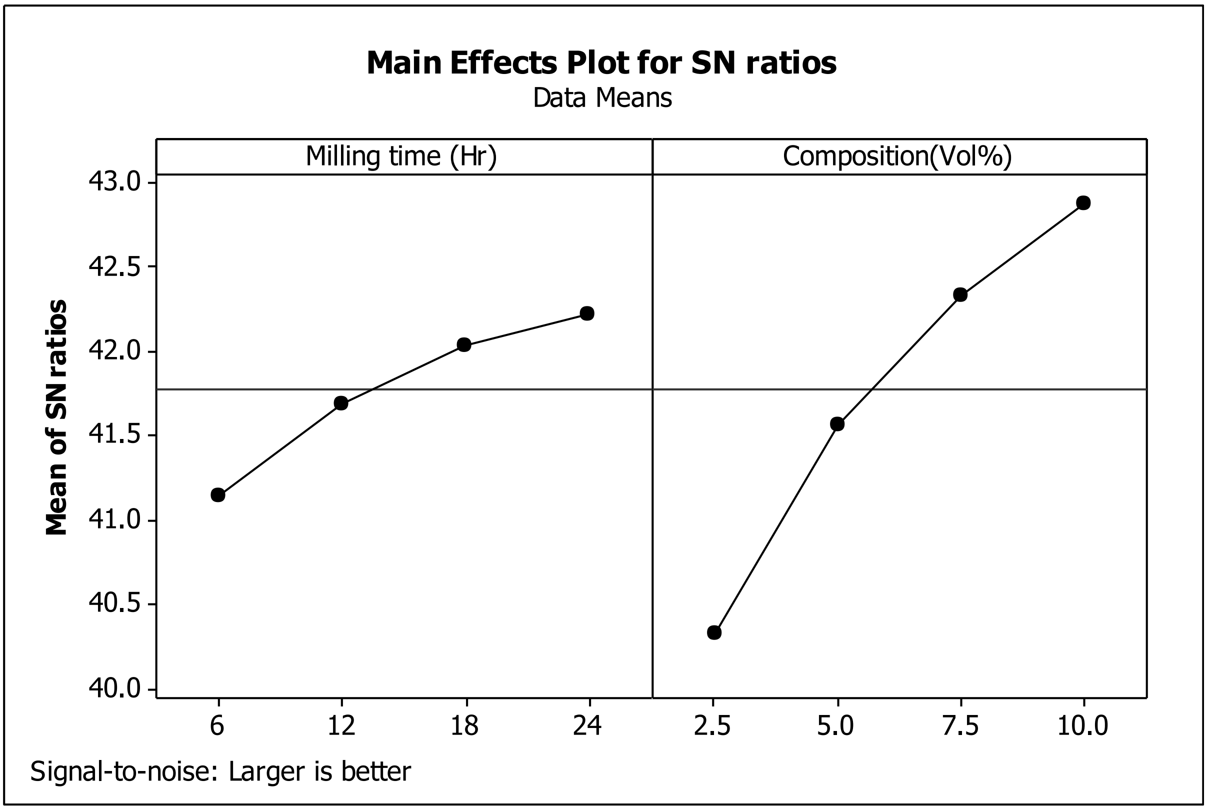

Fig .6 Main effects plot for SN ratios

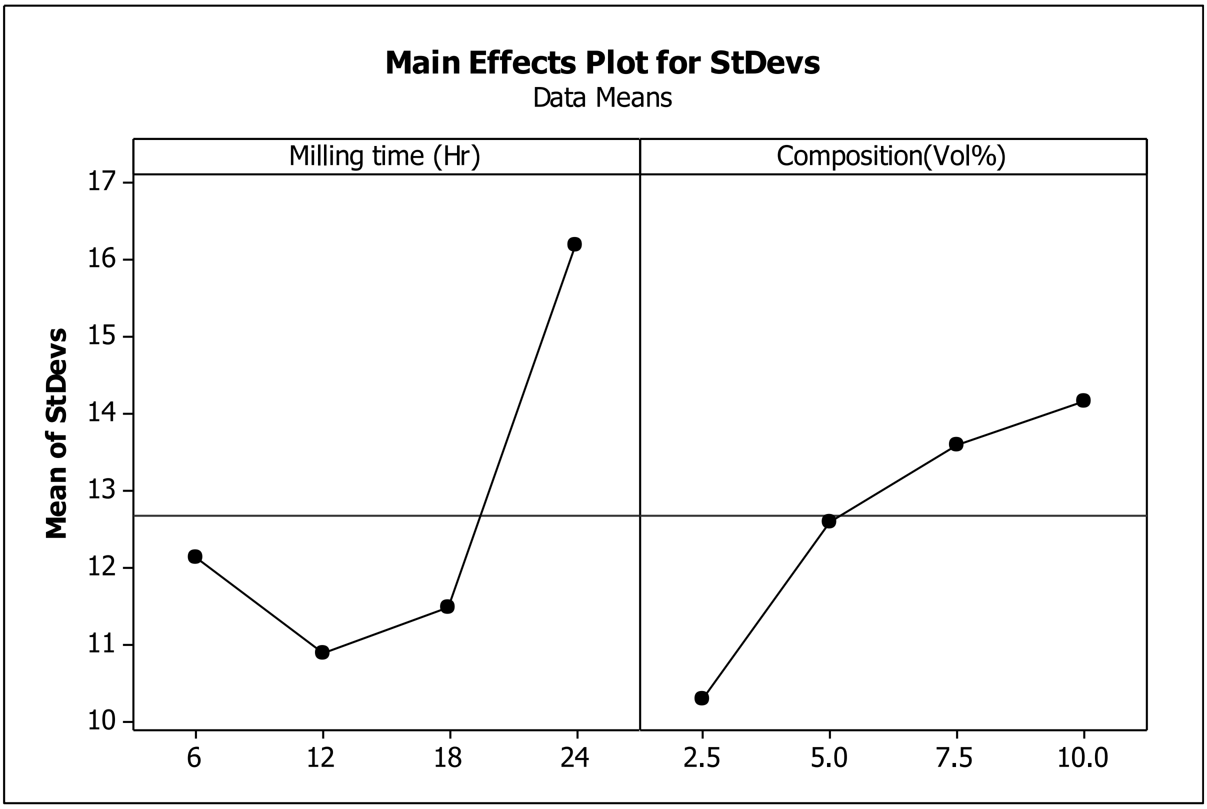

Fig.7 Main effect plot for standard deviation

4.3 Second order regression model

After the factor importance has been determined from the linear statistical model, we move toward process optimization, which requires a regression model of suitable form to account for nonlinearities that exist in the actual relationship between microhardness, milling time and composition. The model can be describes by

$$H=\beta_0+\beta_1 x+\beta_{2X}+\beta_3(xX)+\beta_4(x^2)+\beta_{5(}X^2)+€ \quad \textbf{(3)}$$

Where H represents the microhardness, x represents the milling time, X is the temperature, And β_0- β_5 are unknown regression coefficients can be estimated by using least method squares method.

Significance of x^2 andX^2 in Eq (2) can be determined by calculating their p-values. The p-values were 0.00 and 0.00 respectively. Considering a significance level of 0.05, and based on the p-values approach both factors considered very significant and agrees with the previous conclusions from Fig.5.

R^2 is a parameter represents the amount of variability in the data accounted for by the model. The calculated R^2 value was 59%.This shows a significant increase in the amount of variability in the data. The mean microhardness can be fitted graphically as shown in Fig.8 and 9. Fig.8 shows a three dimensional contour plot for the microhardness as a function of milling time and composition.Fig.9 shows a two dimensional contour plot of constant hardness curves derived from the hardness plot shown in Fig.8

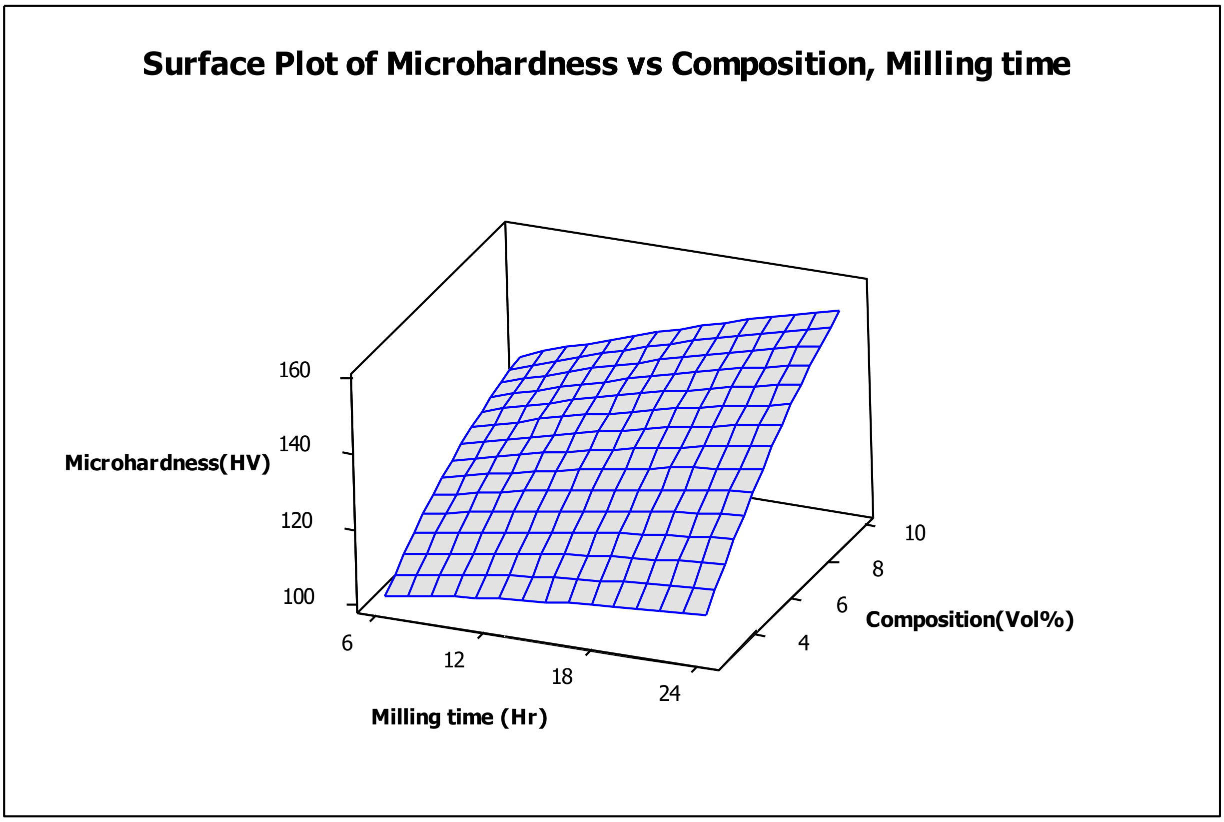

Fig.8 Three dimensional surface plot showing microhardness as a function milling time and composition

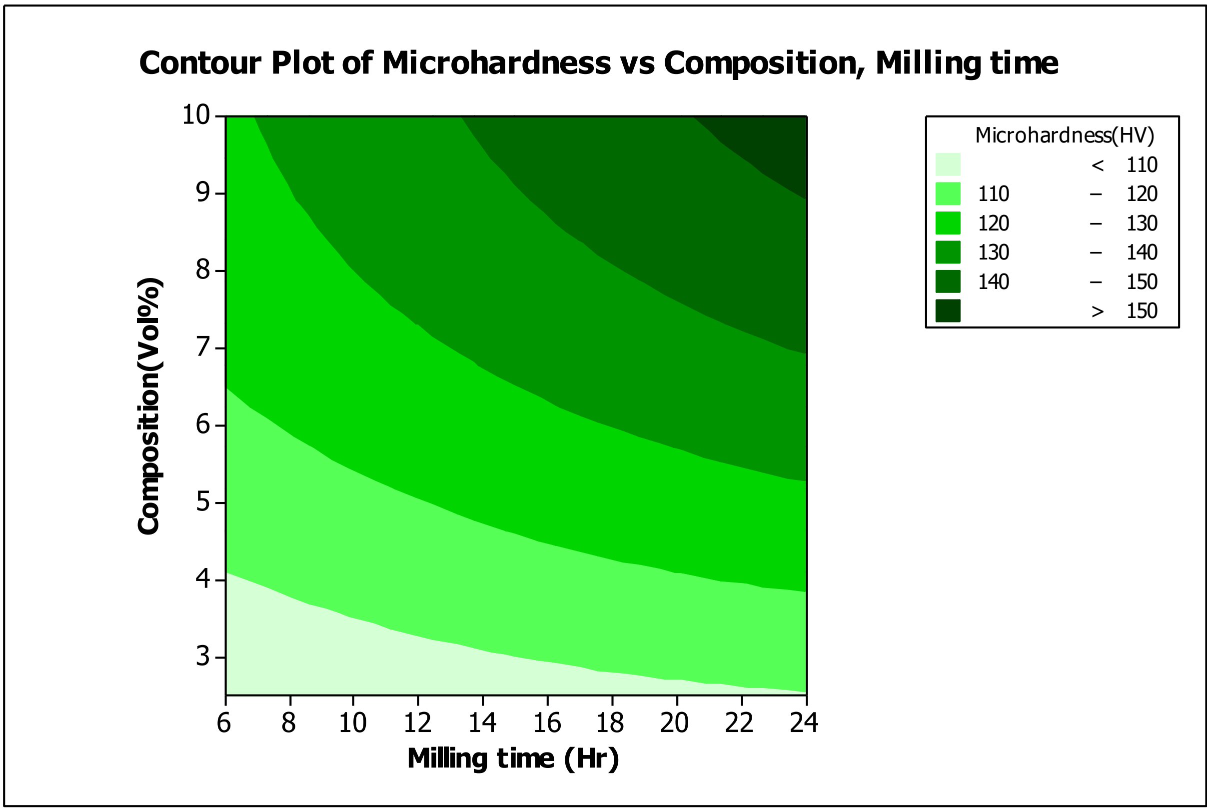

Fig.9 Two dimensional contour plot derived from the surface plot in Fig.8

5. Conclusion

With all the results gotten from the design of experiments analysis presented in this work, we can conclude the following. The results from of the linear statistical model shows that milling time, composition and their interactions factors are all important factors and have significance effect on the microhardness reading of Al-Al2O3 composite. This result was reached by using analysis of variance approach and graphically using the main effect and interaction.

Taguchi method supports the results gained from the analysis of variance with the significance of both the milling time and composition on the microhardness data. With showing preferability to the effect of the composition more than the milling time on the microhardness. Also, indicating that milling time is an adjustable factor used to adjust mean on target.

The second linear model shows some improvements in presenting the relation between microhardness, milling time, and composition in comparison to the linear model by considering and capturing more of the variability in the data.

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Table.1: Tensile properties and microhardness of Al-5vol.-%Al_2O_3- samples produced by powder compact extrusion and powder compact forging of the milled powder.

Material	Yield Strength (MPa)	Ultimate Tensile Strength(MPa)	Plastic strain to Fracture (%)	Microhardness (HV)
Al-5vol.%Al_2O_3 forged	343.7	362.3	8	117
Al-5vol.%Al_2O_3 Extruded	318.2	371.1	8	133

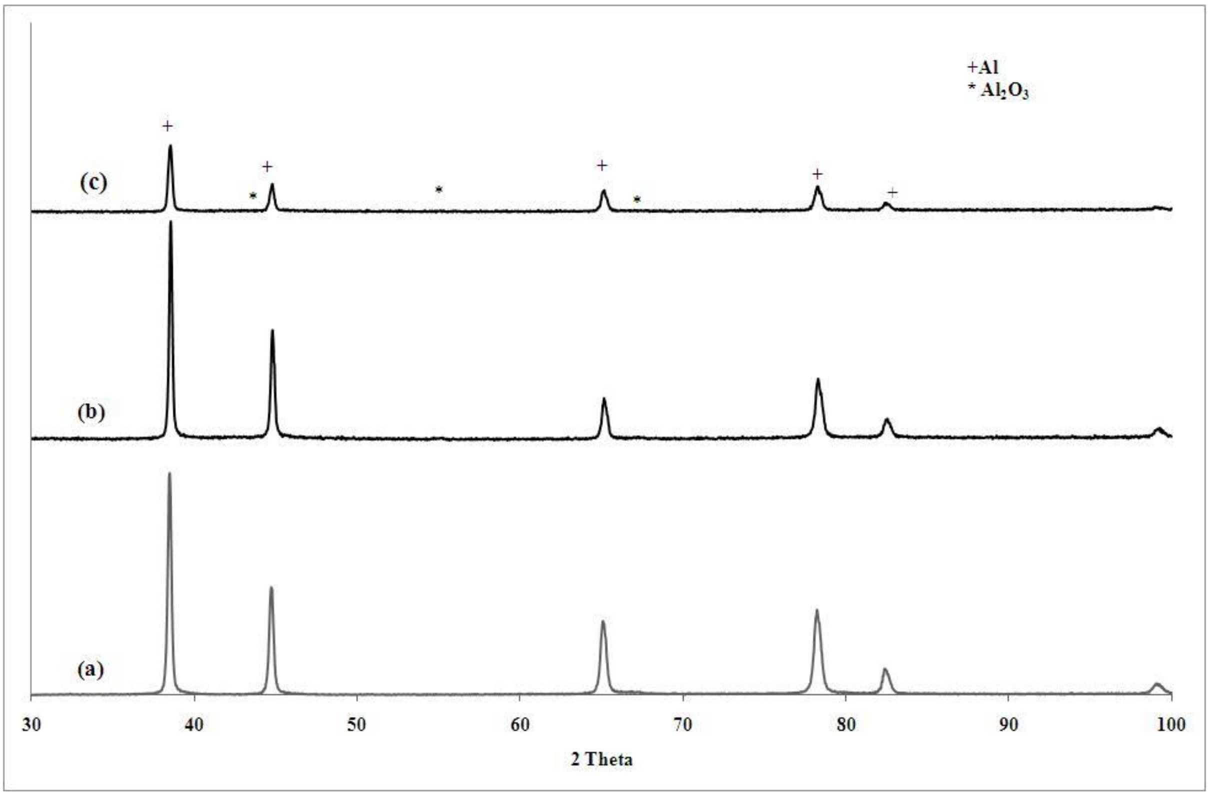


Fig.1: XRD patterns of (a)Al-5vol.%Al_2O_3 nanocomposite powder produced by HEMM; (b) Al-5vol.%Al2O3 nanocomposite disk produced by powder compact forging; (c) Al-5vol.%Al_2O_3 nanocomposite rod produced by powder compact extrusion.

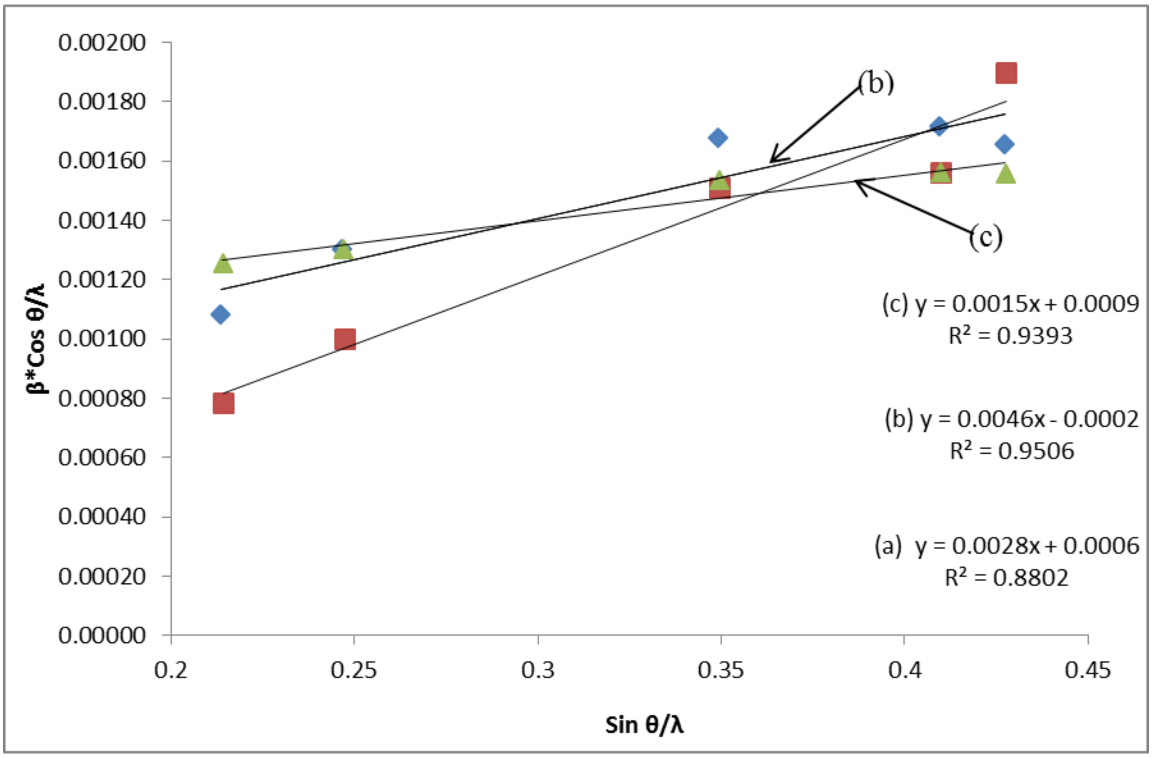
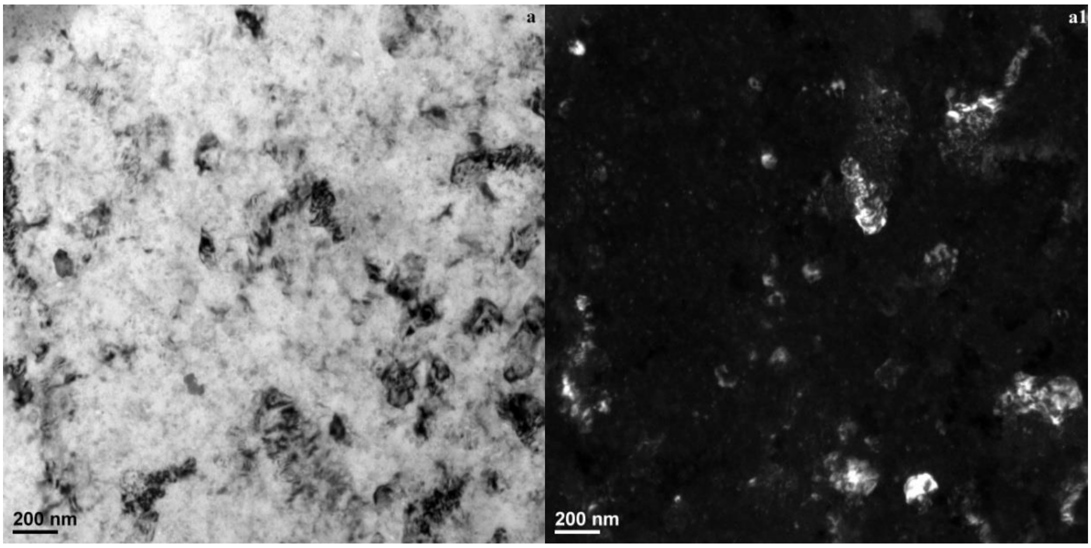


Fig.2: β*Cos θ/λ vs Sin θ/λ plots: (a)Al-5vol.%Al_2O_3 nanocomposite powder produced by HEMM; (b) Al-5vol.%Al_2O_3 nanocomposite disk produced by powder compact forging; (c) Al-5vol.%Al_2O_3 nanocomposite rod produced by powder compact extrusion.



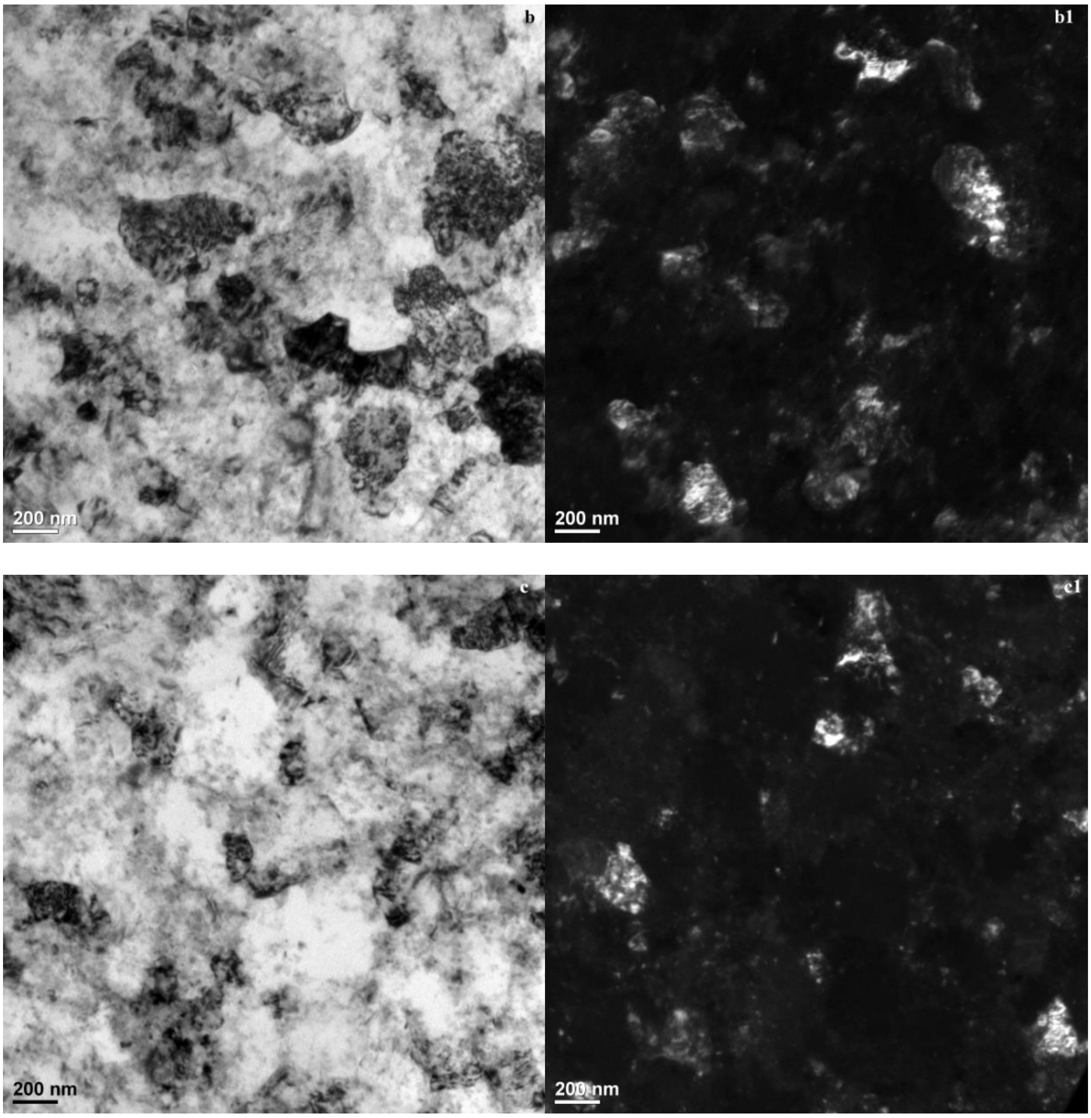


Fig.3: Bright field and dark field TEM images of (a) and (a1) Al-5vol.%Al_2O_3 nanocomposite powder produced by HEMM; (b) and (b1) Al-5vol.%Al_2O_3 nanocomposite disk produced by powder compact forging; (c) and (c1) Al-5vol.%Al_2O_3 nanocomposite rod produced by powder compact extrusion.

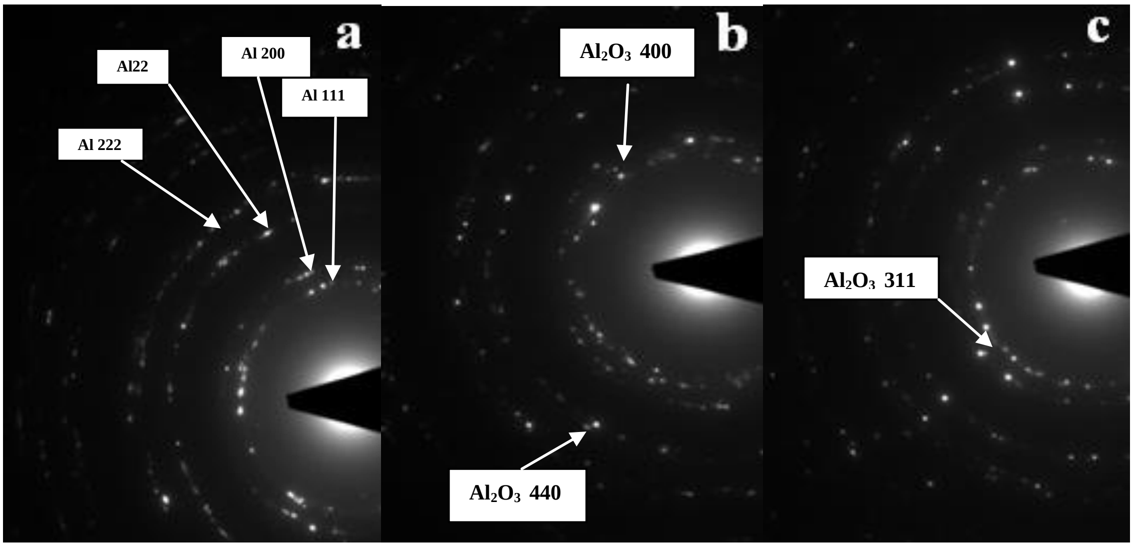


Fig.4: Selected area electron diffraction patterns of (a) Al-5vol.%Al_2O_3 nanocomposite powder produced by HEMM, corrsponding (a) and (a1) in Fig. 3; (b) Al-5vol.%Al2O3 nanocomposite disk produced by powder compact forging, corrsponding (b) and (b) in Fig. 3; (c) Al-5vol.%Al_2O_3 nanocomposite rod produced by powder compact extrusion, corrsponding (c) and (c1) in Fig. 3.

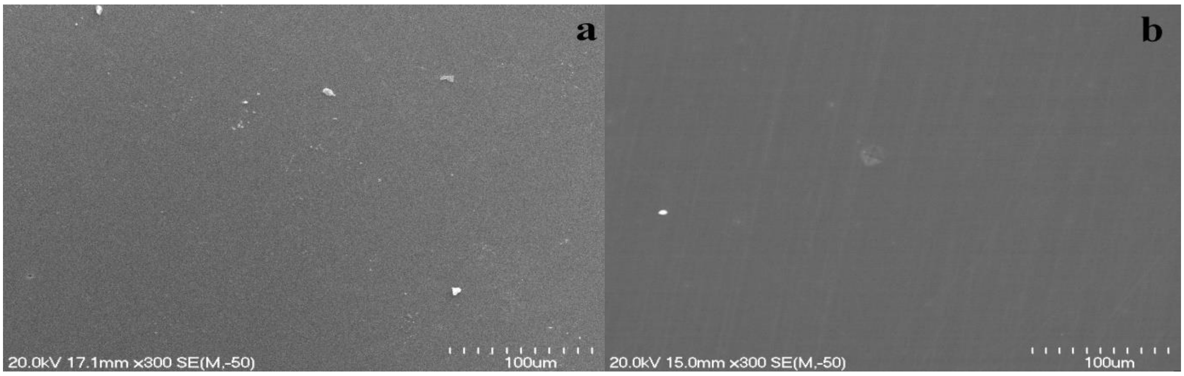


Fig. 5: SEM micrographs of the central part of cross sections of (a) Al-5vol.%Al_2O_3 nanocomposite disk produced by powder compact forging; and (b) Al-5vol.%Al_2O_3 nanocomposite rod produced by powder compact extrusion.

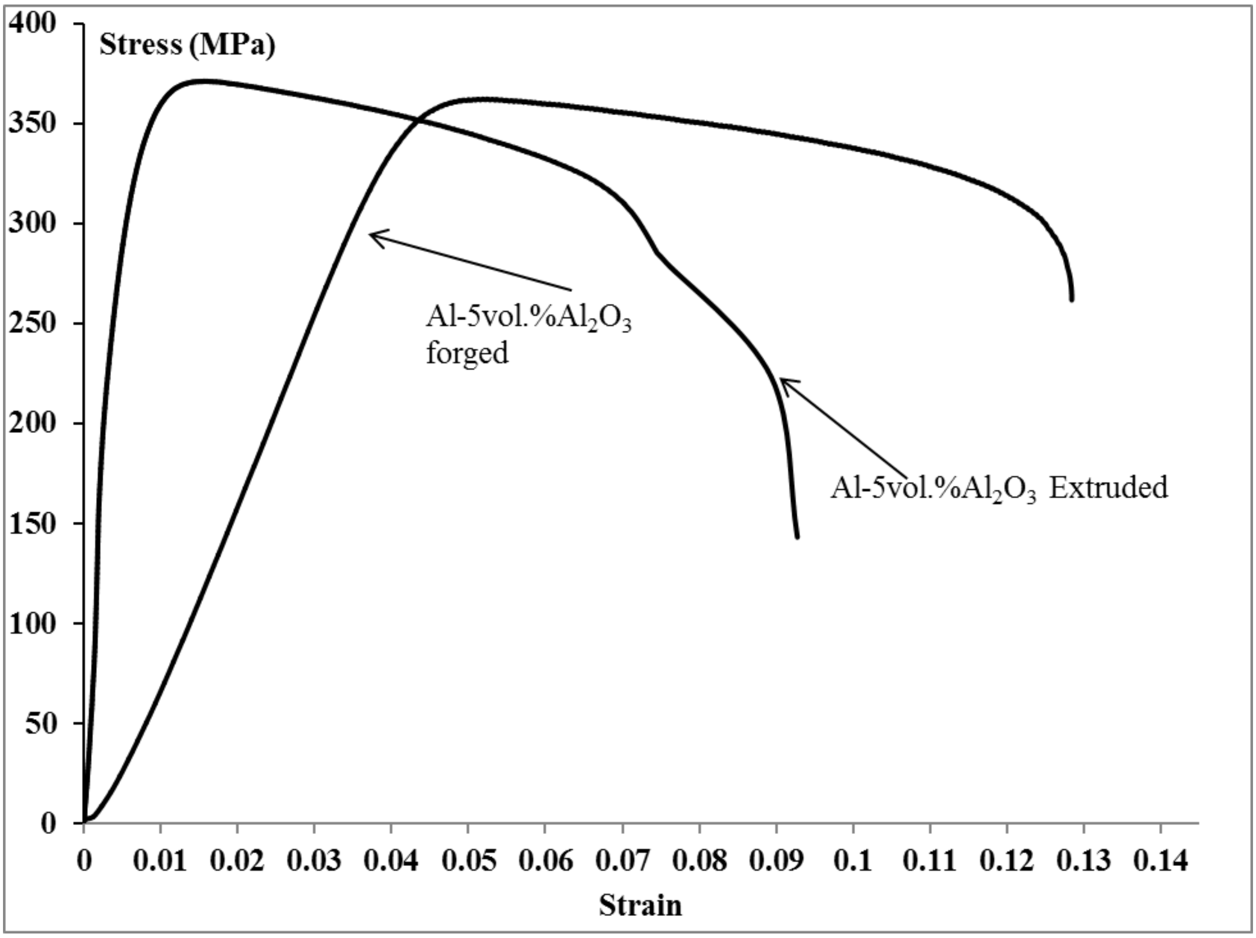
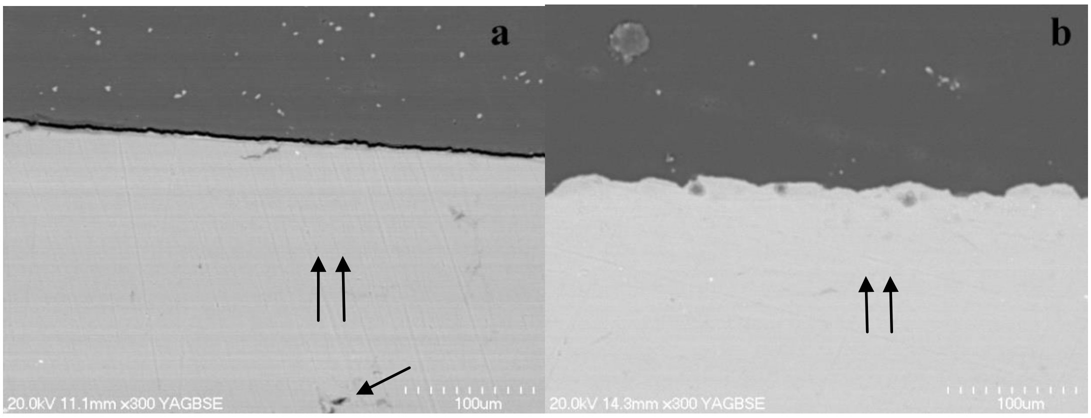


Fig. 6: Tensile engineering stress-strain curves of specimens Al-5vol. %Al_2O_3 nanocomposite specimens cut from Al-5vol.%Al_2O_3 nanocomposite disk produced by powder compact forging and Al-5vol.%Al_2O_3 nanocomposite rod produced by powder compact extrusion, respectively.



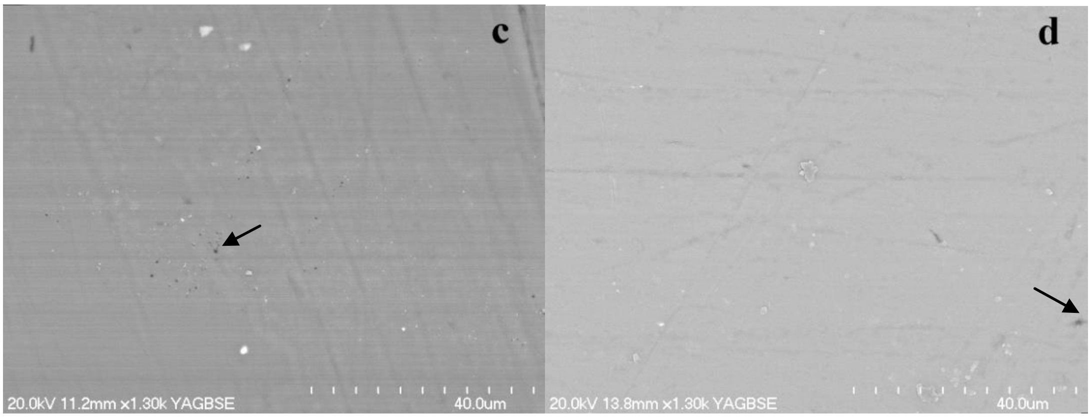


Fig. 7: SEM micrographs of the longitudinal sections of the tensile tested specimens cut from the Al-5vol.% Al_2O_3 nanocomposite samples produced by powder compact forging and powder compact extrusion respectively: (a) just below the fracture surface of the specimen cut from the forged disk; (b) just below the fracture surface of the specimen cut from the extruded rod; (c) away from the fracture surface of the specimen cut from the forged disk but within the gauge length; (d) away from the fracture surface of the specimen cut from the extruded rod but within the gauge length.

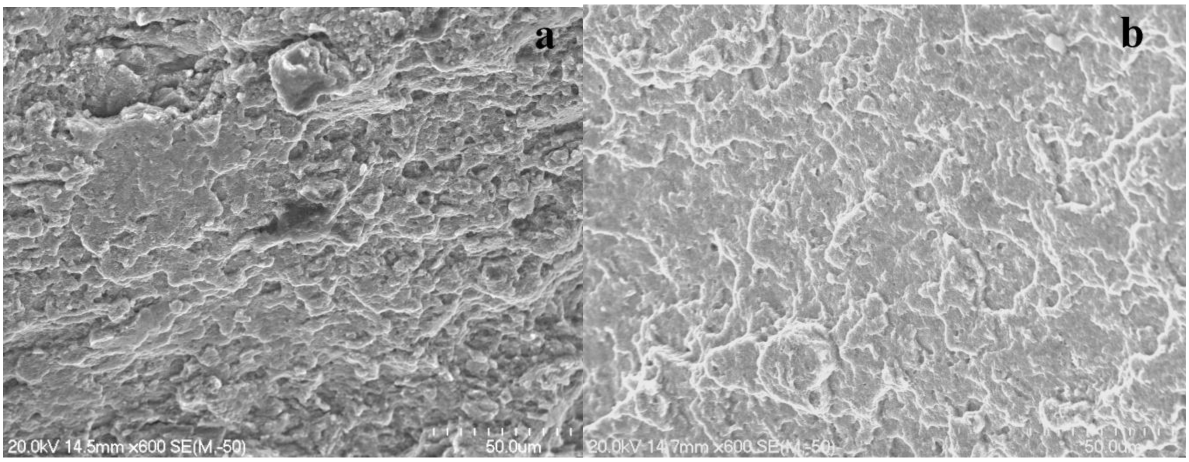


Fig. 8: SEM micrographs of the fracture surfaces of the tensile tested specimens cut from Al-5vol.%Al2O3 nanocomposite samples produced by (a) powder compact forging and (b) powder compact extrusion, respectively.