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## **A NOVEL TECHNIQUE FOR PRODUCTION OF FRICTION STIR PROCESSED RHEOCAST NANO REINFORCED AA2024 ALLOY**

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### **ABSTRACT**

Rheocast Al 2024 alloy and rheocast Al 2024 / Al<sub>2</sub>O<sub>3</sub> nano composite were friction stir processed at constant parameters, i.e. rotation speed of 1000 rpm and linear speed of 20 mm/min. The microstructure of the as-cast and FSPed alloys were examined by optical and scanning electron microscopy carried out to evaluate the effect of FSP on grain refinement. Microhardness revealed that the semi-solid state processing does not allow to obtain an even dispersion of nanoparticles within the matrix; a secondary process such as friction stir processing, on the contrary, allows both to homogenize the microstructure and to eliminate casting defects associated with low particle wettability (porosities and particle agglomeration), leading to an enhanced exploitation of nanoparticles strengthening effect.

### **KEY WORDS**

Nanocomposite, Friction Stir Processing, AA2024 alloy, semisolid processing

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## INTRODUCTION

Compcasting route is a rheocasting process that involves the injection of reinforcement particles into semisolid state alloys. Compcasting is generally thought to be a processing route allowing to obtain quite uniform distribution of reinforcing particles, as well as to enhance particle wettability [1].

El-Mahallawi et al. [2-3] studied the influence of nano dispersions on mechanical properties of semisolid Al casts using nanoparticles as reinforcement agents, results showed enhancement in the tensile strength reached to about 195 MPa of the Al<sub>2</sub>O<sub>3</sub> nano-dispersed alloys, accompanied by significant increase in the elongation percentage, supported by evidence of refined structure.

Dispersion of nanosized reinforcements on metallic substrate to produce surface metal matrix nanocomposite (SMMNC) and the control of its distribution are difficult to achieve by conventional surface treatments [4].

Mahdy [5] shows that the primary solid particles already formed in the semi-solid slurry can mechanically entrap the reinforcing particles, prevent their gravity segregation and reduce their agglomeration, these result in better distribution of the reinforcement particles.

FSP leads to localized severe plastic deformation of a material and thus to homogenization of microstructure and grain refinement, thereby resulting in improved material properties. Previous work has studied the effects of FSP on commercial aluminum alloys [7-11]. FSP was used for microstructure modification of cast aluminum alloys to enhance their mechanical properties; also FSP of alumina particle reinforced aluminum alloys has been studied [12-13]. Ahmed, et al. [14, 15] developed AA7075/nano-alumina composite using FSP; the results showed that FSP can be utilized to develop surface-nanocomposites with Al<sub>2</sub>O<sub>3</sub> nanodispersions embedded in the matrix. The new surface nanocomposites showed enhancement in the hardness of the surface of A7075 to almost double of the starting material.

Thus the aim of the present work is to fabricate sound nanocomposite of Al 2024/ Al<sub>2</sub>O<sub>3</sub> using rheocast and friction stir processing, also study effect of FSP on grain refinement of the matrix and alumina nanodispersion in Al 2024/ Al<sub>2</sub>O<sub>3</sub>, which could be enhanced by mechanical properties.

## MATERIALS AND METHOD

An AA2024/Al<sub>2</sub>O<sub>3</sub> nanocomposite was produced by compocasting, allowing the dispersion of 1 wt% Al<sub>2</sub>O<sub>3</sub> nanoparticles into the matrix at the semi-solid state through mechanical stirring. Chemical composition of the matrix and properties of the reinforcing particles are reported in Table 1 and 2, respectively.

A charge of 400 g of Al 2024 wrought alloy was melted in an electric heat resistance furnace, using a carbide crucible, and a three-blade graphite stirrer driven by speed motor of 800 rpm. After reaching the liquid state the melt was degassed with hexachlorethane degasser tablets, to get rid of gases. At this temperature (630 °C) the

reinforcing Al<sub>2</sub>O<sub>3</sub> particles were introduced into the slurry. The nano Al<sub>2</sub>O<sub>3</sub> particles were previously treated using high energy ball mill for about 5 minutes in order to avoid particle clustering, then packaged in aluminum foil (1% of charge weight). The packages were preheated for 1 hour at 200°C before being introduced to the molten aluminum alloy. Another charge of Al 2024 wrought alloy was melt at same conditions but without adding reinforcement. The melt (either unreinforced or reinforced) was cast in a stainless steel mould with dimensions of 250 mm length, 50 mm width and 10 mm thickness, producing as cast Al 2024 and nanocomposite AA 2024/Al<sub>2</sub>O<sub>3</sub> plates.

Friction stir processing (FSP) was applied on the cast materials; monolithic & nanocomposite, using controlled FSW/FSP machine located at Suez University. The process was carried out using a cylindrical FSP tool made of H13 tool steel heat-treated to obtain 58HRC hardness. The FSP tool dimensions were 6 mm probe diameter, 6 mm probe length and 20 mm diameter shoulder, rotation speed of 1000 rpm, traverse speed of 20 mm/min, and tool the tilt angle was set at 3°. Examples of the AA2024/Al<sub>2</sub>O<sub>3</sub> nanocomposite plates before and after FSP are shown in Fig. 1.

Macrographs of the cross sections of samples were obtained through a multifocal microscope (Hirox). In order to carry out microstructural analyses, metallographic samples were prepared for examination by using optical microscopy (OM) and scanning electron microscopy equipped with energy dispersive spectroscopy (SEM-EDS) to assess microstructural refinement induced by FSP.

The investigated materials were measured at ambient temperature by Vickers microhardness profiles. Vickers microhardness measurements were performed on the cross section of samples, under load of 4.9 N and for a dwell time of 10s. 20 microhardness indentations were performed every 500 µm at different depth from the FSP surface, namely 2, 4 and 6 mm, as shown in Fig. 2.

## RESULTS AND DISCUSSION

### Microstructural Analyses

The macrostructure of as cast material reveal fine structure as shown in Fig.3-a, while metal matrix composites structure contains pores and particles clusters as shown in Fig.3-b, this was expected, as widely reported by several investigators [2-6], It arises due to gas entrapment, during mixing, hydrogen evolution and shrinkage, wettability issue between reinforcement and matrix alloy increasing tendency for agglomeration of particles [6]. It was reviewed that porosity levels should be kept to minimum as this kind of a composite defect can be detrimental as it can control mechanical properties of the cast metal.

Macrographs of the FSP processed unreinforced alloy and nanocomposite are reported in Fig.3 (c, d). The FSP zones are characterized by different interfaces between the nugget and the TMAZ, at the advancing side (AS) and retreating side (RS). It is also possible to observe a slight difference in the shape of the nugget zone, between the unreinforced matrix and the nanocomposite that the rotation speed in composite reinforced with nanoparticles is hindered by the nanoparticles clustering.

Optical micrographs of the as-cast unreinforced alloy before and after friction stir process are shown in Fig. 4. The conventional cast microstructure shows primary  $\alpha$ (Al) dendrites surrounded by the eutectic structure, with the intermetallic compounds consisting of  $\text{Al}_2\text{Cu}$  and  $\text{Al}_2\text{CuMg}$  as shown in Fig.4 (a, b). Absence of cavities may be observed after applying FSP inside the nugget zone as shown in Fig. 4-c and also near advance sided as shown in Fig.4-d. This should be related to the thermomechanical action of the pin, which induces plastic deformation of the matrix, therefore eliminating voids and cavities [7-9].

The structure of the compocast AA2024/ $\text{Al}_2\text{O}_3$  before FSP is shown in Fig. 5 (a, b), this structure was changed after mechanical stirring and nanoparticles addition to be spheroidized. Some regions still showing dendritic structure. The  $\alpha$ -dendrites are characterized by a quasi-globular morphology, more accentuated in the composite. Similar behavior has been reported, supporting that the addition of  $\text{Al}_2\text{O}_3$  nano-particles to aluminum alloys has reduced the grain size of the aluminum matrix [2-4, 6]. A significant grain size reduction in the stir zone has occurred relative to the cast material grain structure, as shown in Fig. 5 (c, d). In addition, the nano particles cluster size is reduced and the reinforcement distribution is improved. Breaking the intermetallics formed at casting and further separation of particles from the clusters during FSP resulted in improved nanoparticle distributions.

Intermetallics percentages of cast AA2024/ $\text{Al}_2\text{O}_3$  was reported by SEM-EDS analyses as shown in Fig. 6 (a, b). The nanoparticles at the cavities and pores as shown in Fig. 6 (c, d, e), which are the last parts to solidify, has been explained by the tendency of these alloys to exhibit a pushing mechanism for the foreign particles, rather than the engulfment mechanisms shown when the particles are homogeneously incorporated within the grain boundaries during solidification. It has been shown that as the slurry of the melt contains more liquid phases remaining and when the heat extraction by the die is non-uniform, some irregular particles are produced at the cavities as shown in the microstructure in Fig.6 [16].

SEM-EDS analyses of the friction stir processed samples are presented in Figures. 7 and 8. A very fine grained microstructure, due to the dynamic recrystallization process acting during FSP, was observed in the stirred zone of both unreinforced alloy and nanocomposite alloy. This implies that the FSP parameters used resulted in sufficient heat input and proper material flow for producing sound stir zone. This can be attributed to the change in the contact condition of the FSP tool with the surrounding material from sticking condition at low rotation speed to slipping condition at high rotation speed due to the increase of the heat input [14]. Inducing localized severe plastic deformation, led to  $\alpha$ -Al dendrite fragmentation as a further consequence of the pin action, size and aspect ratio of the intermetallics decreased significantly, as can be clearly seen by comparing Fig.6 c and d. This can be mainly related to the mechanical fragmentation, but also to the elevated temperature induced by the pin rotation, which could partially dissolve the intermetallic particles [14, 16].

### **Micro-Hardness**

Microhardness profiles of the FSP unreinforced alloy and nanocomposite are shown

in Fig.9. The profiles were carried out at different depth from processed surface, namely at 2, 4 and 6 mm. The reference microhardness value was measured on the unprocessed base material of the unreinforced alloy (93 HV) and nanocomposite (95 HV). Hardness values variation should be related to the grain refinement induced by the process, leading to an enhancement of mechanical properties. The relatively lower micro hardness in TMAZ could have resulted from the large grain size and the dissolution of precipitates into the matrix during FSP [18-19].

Comparing profiles of FSP unreinforced alloy and nanocomposite, it is possible to observe a remarkable increase in microhardness in the unreinforced alloy, where peak values of about 140-155 HV were measured (for reference, 93 HV in the base material). This behavior was observed at 2 and 6 mm depth. It is reasonable to relate this microhardness enhancement to a more even distribution of nanoparticles in AA2024/Al<sub>2</sub>O<sub>3</sub> composite. As previously observed by optical microscopy, the microstructure of AA 2024 was characterized by a more turbulent plastic flow, which could have helped in homogeneous particle distribution.

Also hardness maps shown in Fig.10, illustrates that localized surface effect of friction stir processing take place and so hardness values are so high near to surface of the nano samples this may be due to nano particles restriction to processing, otherwise hardness map of base matrix shows more homogenous of hardness distribution in nugget center. Results show that there is a drop of hardness values in nugget zone of AA2024/ Al<sub>2</sub>O<sub>3</sub> compared to that of AA 2024, these results are in agreement with the results reported by A. Denquin, et. al. [20] and T. Nelson, et.al. [21]. The hardness drop at the heat affected zone is due to the fact that there is no mechanical deformation (stirring) at that zone; however the peak temperature reached is enough to soften the material near the nugget. nano particles used this soften regions to make it more hard but high FSP rotation rate doesn't give it chance to occupy this region so high hardness values were obtained at low rate of speeds and on same direction of rotation which is from advancing side to retreating side.

## **CONCLUSION**

The present investigation confirmed the beneficial effects of nanoparticles on aluminum alloys mechanical behavior if homogeneously distributed. In this regard, friction stir processing resulted to be a promising method for the production of nanocomposites characterized by high levels of hardness.

## **ACKNOWLEDGEMENT**

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**Table 1.** Chemical composition (wt. %) of Al 2024 matrix

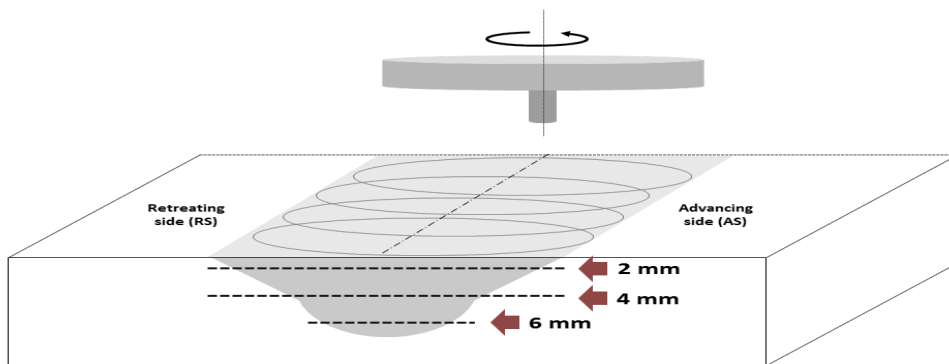
Element	Cu	Mg	Mn	Fe	Si	Zn	Cr	Al
wt%	4.39	1.26	0.57	0.50	0.50	0.25	0.10	Bal.

**Table 2.** Characteristics of the reinforcing Al<sub>2</sub>O<sub>3</sub> nanoparticles

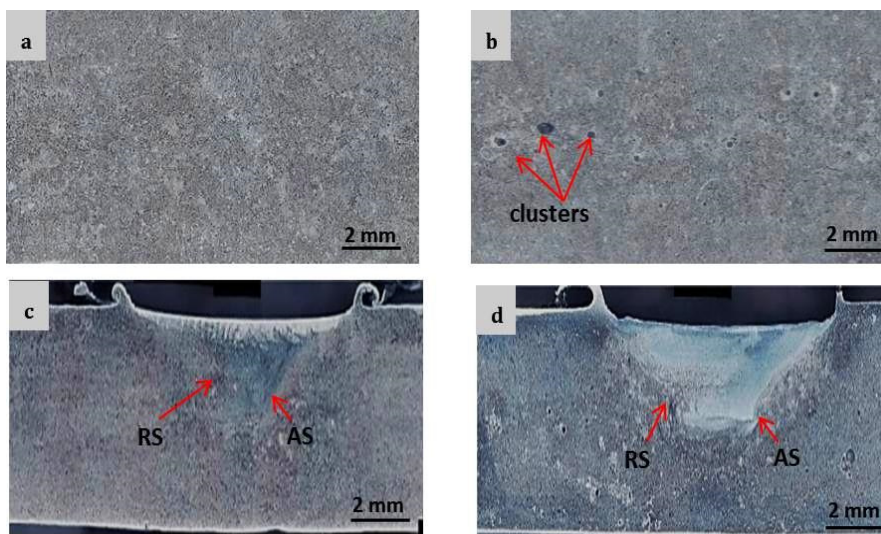
Reinforcement	Density (g/cm <sup>3</sup> )	Structure	E (GPa)	Av. Size (nm)	Melting point
γ-Al <sub>2</sub> O <sub>3</sub>	3.60	FCC	380	50	2054 °C



**Fig.1.** Example of semi-solid processed AA2024/Al<sub>2</sub>O<sub>3</sub> nanocomposite plate.

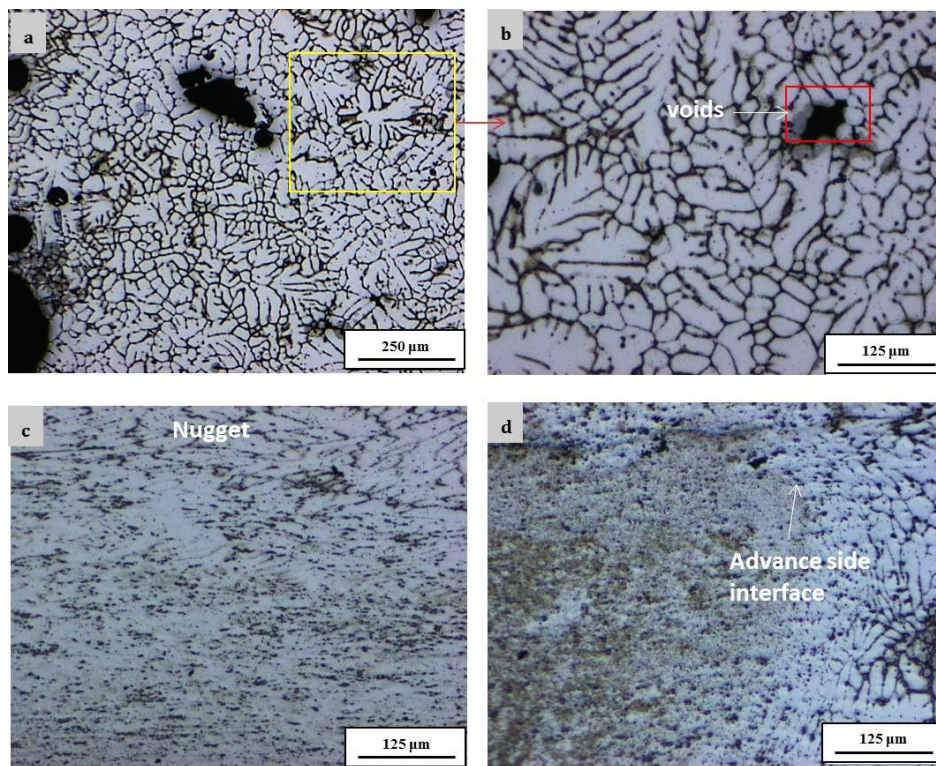


**Fig.2.** Schematic illustration of microhardness profiles.

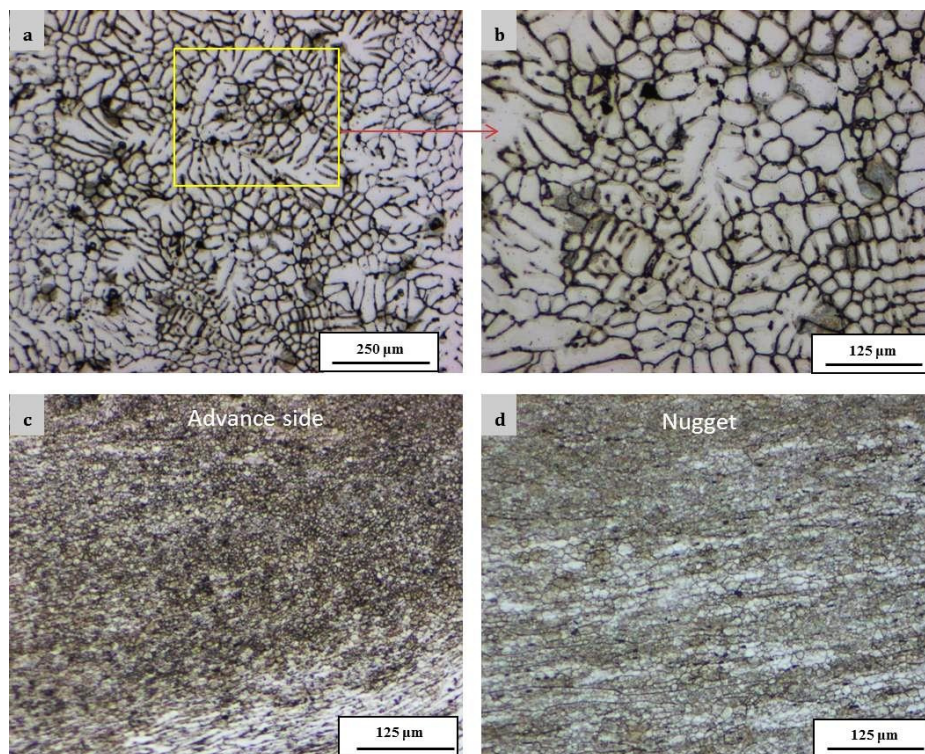


**Fig.3.** Macrograph of (a) cast AA 2024,(b) cast AA2024 /Al<sub>2</sub>O<sub>3</sub>, (c) FSPed AA 2024 and (d) FSPed AA2024 /Al<sub>2</sub>O<sub>3</sub>.

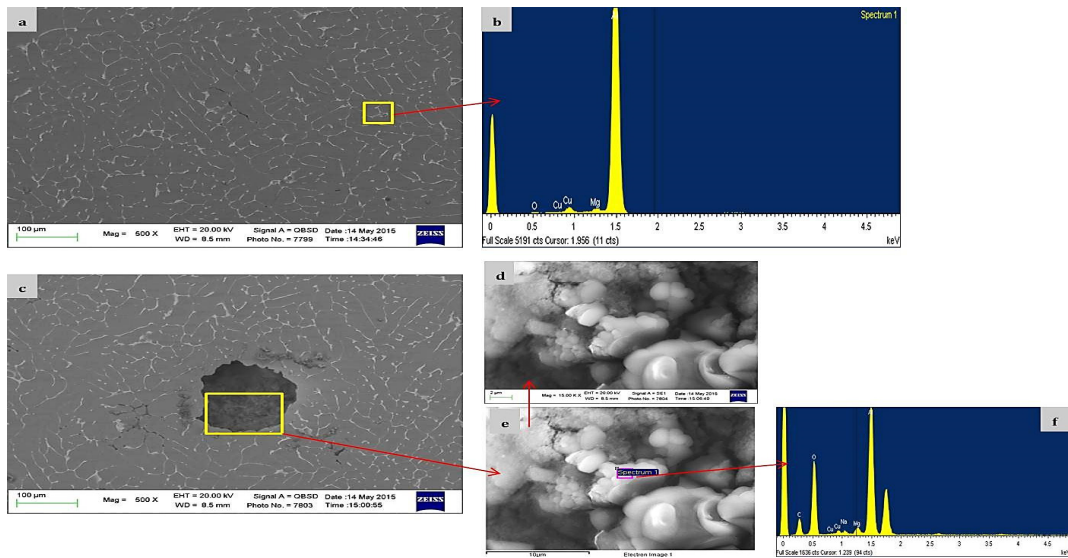




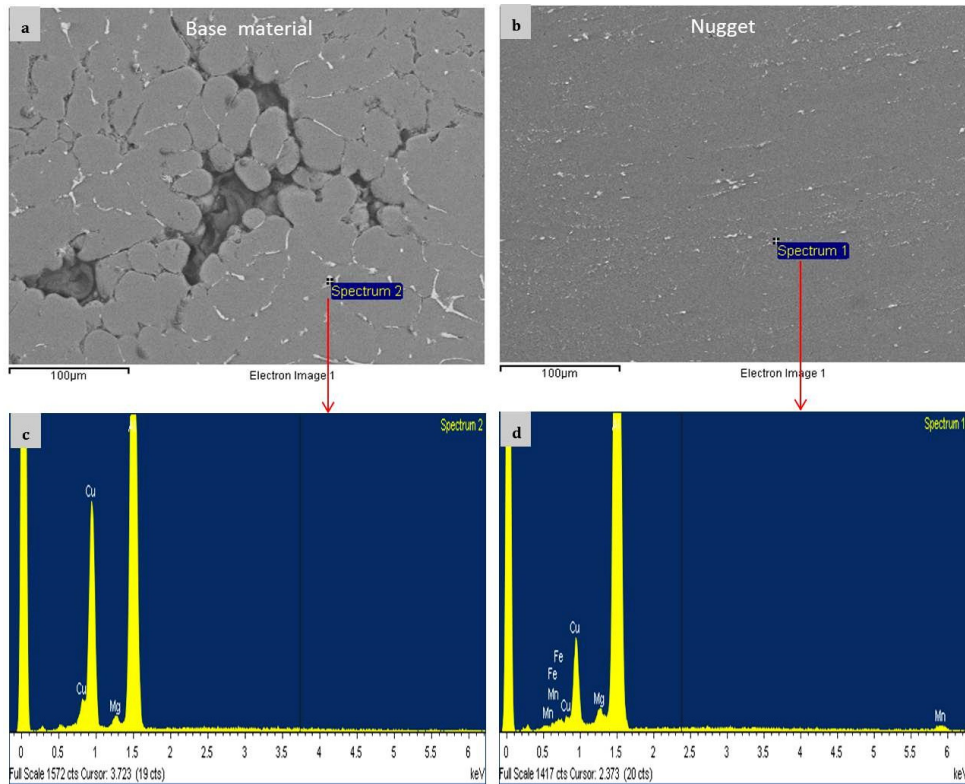
**Fig.4.** micrographs of AA2024 before FSP (a, b) and after FSP (c, d).



**Fig.5.** Micrographs of rheocast AA2024 /Al<sub>2</sub>O<sub>3</sub> before (a, b) and after FSP(c, d).

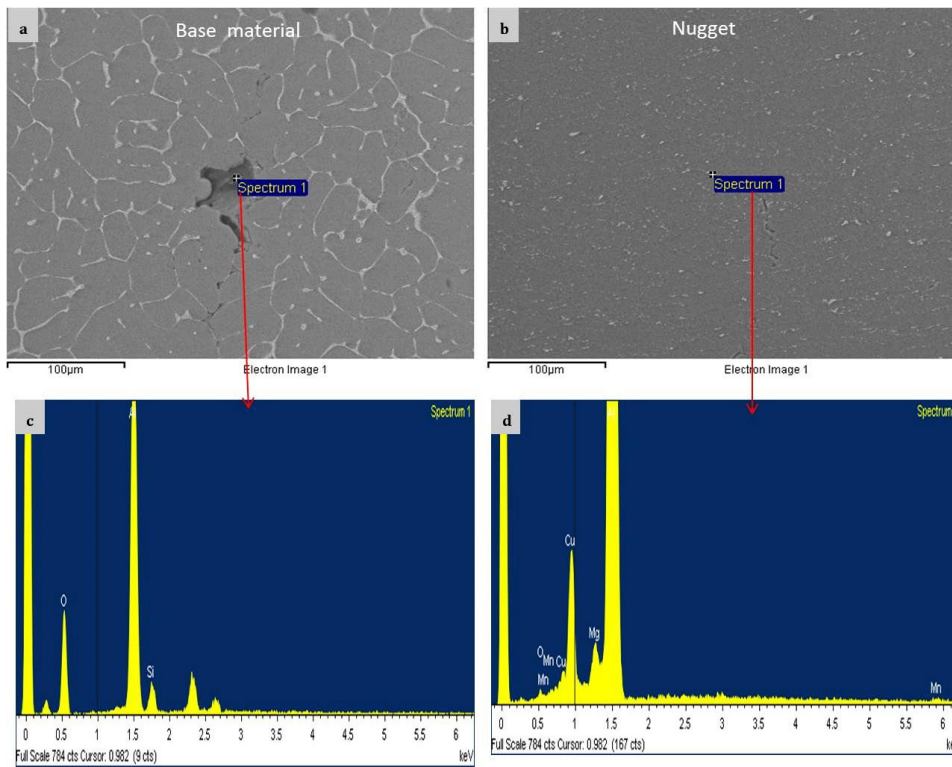


**Fig.6.** SEM micrographs of AA 2024 (a) and EDS pattern (b), also porosity revealed in rheocast AA 2024/ Al<sub>2</sub>O<sub>3</sub> (c), contains morphology of alumina (d, e), EDS pattern confirm Al<sub>2</sub>O<sub>3</sub> existence (f).

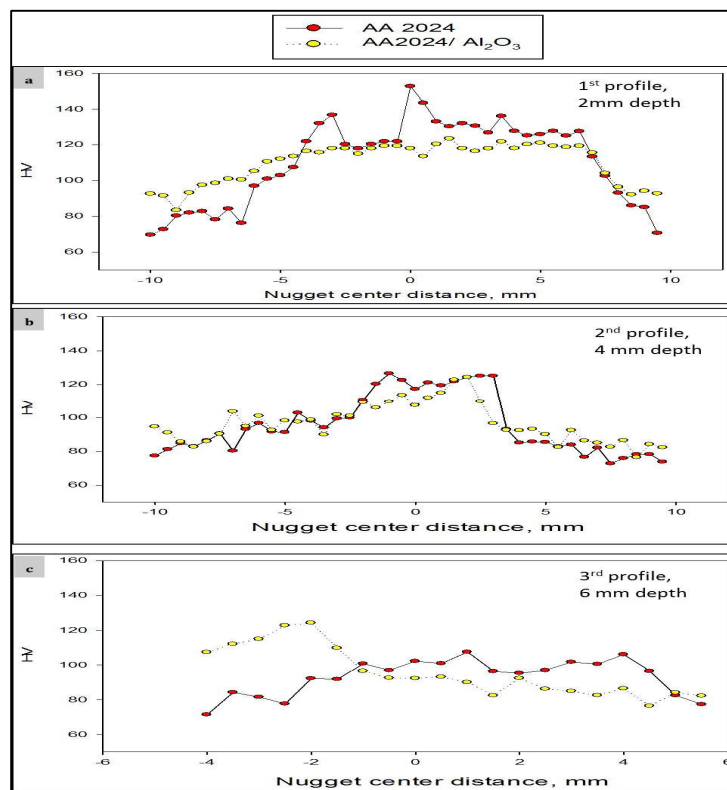


**Fig.7.** Comparative SEM micrographs of AA 2024 base (a,c) and nugget zone (b,d).

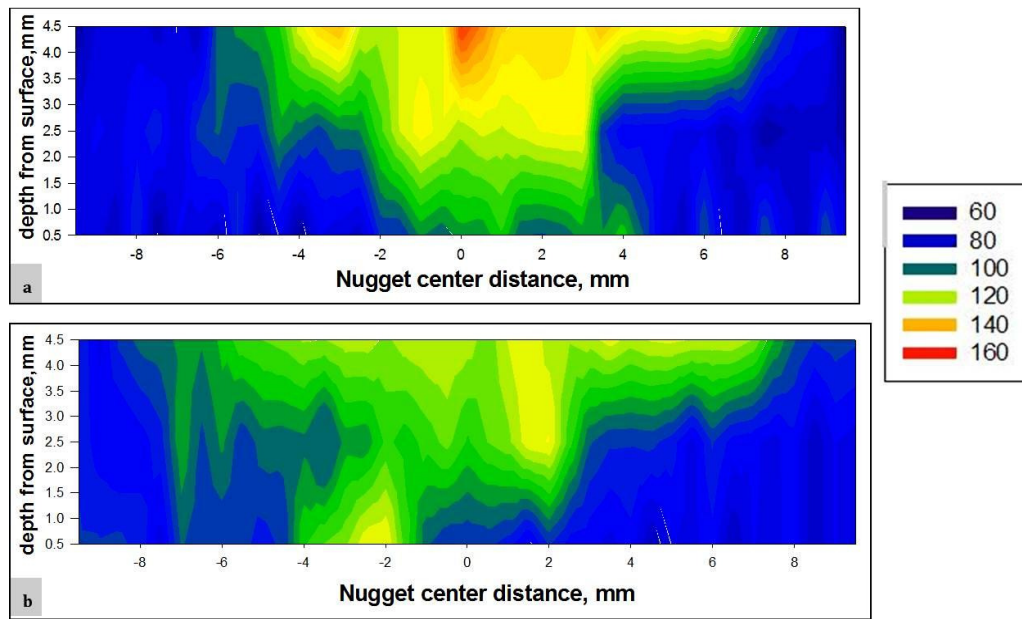




**Fig.8.** SEM micrographs of AA 2024/  $Al_2O_3$  (a), and NZ structure (b) EDS patterns (c, d).



**Fig.9.** Hardness profiles of FSPed AA2024 and AA2024/  $Al_2O_3$ .



**Fig.10.** Microhardness maps of FSPed AA2024 (a) and AA2024/ Al<sub>2</sub>O<sub>3</sub> (b).