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Umida Ziyamuhamedov
Tashkent State Transport University, 100067, st.Adilkhodjaev 2, Tashkent, Uzbekistan, ulugbekovich0510@gmail.com

M Wani
Tribology Laboratory, Mechanical Engineering Department, National Institute of Technology Srinagar, Hazratbal, Kashmir-190006, J&K, India

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DESIGN AND FABRICATION OF ALUMINUM BASED WS_2 NANO-COMPOSITE THROUGH POWDER METALLURGY

M. F. Wani[1], U. Ziyamukhamedova[2]
[1]*Tribology Laboratory, Mechanical Engineering Department*
National Institute of Technology, Srinagar, Hazratbal, Kashmir-190006, J&K, India
[2]*Tashkent State Transport University, 100067, St. Adilkhodjaev 2, Tashkent, Uzbekistan*
[2]Yeoju technical institute in Tashkent, 100121, St. Usman Nasir 156, Tashkent, Uzbekistan

Abstract: *In this research paper, experimental studies on the fabrication of Aluminum based metal matrix composites composed are presented. The Aluminum based metal matrix composite was developed with varying percentage of WS_2. Physical and mechanical properties were measured to assess the influence of addition of WS_2 on density and micro hardness of composite*.

Keywords: *metal matrix composite (MMC), Ceramic matrix composite (CMC), coat, optical microscope (OM), scanning electron microscope (SEM), aluminum*

INTRODUCTION. Development of materials for enhancing efficiency of mechanical system for various engineering applications is one of the utmost task of engineers and scientists. Various materials, like metal matrix composite (MMC), Ceramic matrix composite (CMC) and advanced coatings have already been developed to serve under extreme conditions. These materials and their composites are used in various components and systems in aerospace, automobile, electrical and structural industries. Among various types of metals, aluminum is one of the most commonly used for the production of MMCs that has gained special interest in the field of automobile and aerospace industries [1-2]. MMCs of Al alloy matrix modify the properties of the composite as desired by designer suitable for typical applications. Improvements in hardness, tensile strength, wettability, friction properties, wear resistance or load carrying capacity is achieved, depending on the type of particle constituents [1–3].

The extent of the improvements corresponds to an application-specific optimal particle size [1,4]. Uniform particle distribution in the Al matrix and an adequate concentration provides the deserted properties of the composite [4,7-12]. The inferior properties of pure aluminum, has lead to ceramic particle additions such as SiC and Al2O3 to Al MMCs to increase the mechanical properties of the Al matrix [1,5,6,10]. But their addition leads to a high coefficient of friction and their abrasiveness in the contact above a certain applied load [11]. To mitigate this aspect, particles such as graphite or tungsten and molybdenum chalcogenides are added to reduce friction in tribological contacts due to the very low shearing forces between their weakly bonded layers of atoms. Thereby the development of aluminum MMCs dispersed with solid lubricants is primarily directed towards overcoming the principle drawbacks of aluminum as a tribological material.

Tungsten disulfide, one of the novel materials, has shown excellent performance by reducing friction and wear in composites with different matrices and its individual effect on aluminum metal, following press and sinter powder metallurgy route has not been evaluated yet. Further in applications involving vacuum or dry environments WS_2 is known to perform better than graphite [13]. The conventional press-and-sinter powder metallurgy (P/M) technique is a unique cost-effective method for net shape or near net shape fabrication of complex aluminum parts.

Although aluminum powder is readily available, aluminum P/M unfortunately remains a minor participant in the overall aluminum powder market. Similar to the development of any other manufacturing industry, the future of aluminum P/M will largely depend on more value-added innovative designs and products and improved productivity. Further sintering of aluminum composites is a challenging field and evaluating isolated properties of the composite itself, far challenging and thus a research pattern that others do not follow generally. WS_2 has a crystal structure which is composed of layers held by weak inter layer Vander Waals. Bonding between S-S and W-W is covalent and between W-S is covalent too.

The sandwich -like structures are held by weak Vander Waals forces [14]. Excellent erosion and corrosion resistances and High reactivity in humid environment [15]. Formation of thin coating during wear that helps to increase durability and wear resistance Higher lubricity than Graphite, MoS_2 [16-18]. After exhaustive literature survey it was found that although many composites were fabricated with Al and Tungsten Disulphide along with reinforcements through powder metallurgy route, but isolated composites of Al and nano tungsten disulphide have not been evaluated.

Composites with nano WS_2 as a solid lubricating agent via simple powder metallurgy route has not been explored yet for general bearing application. It has been seen that WS_2 coatings work out well for a specific period of time, but using WS_2 as an additive replenishes the layers after every cycle and thereby increasing the life. Also effect of various percentages (2,4,6%) of WS_2 on aluminum composite has not been evaluated to elucidate the mechanisms of friction and wear over a broader spectrum.

Thus the objective of the present study is to fabricate an aluminum MMC with varying percentages of Nano WS_2 by powder metallurgy route (press and sinter). Although composites based on an aluminum alloy rather than pure aluminum would display superior properties for the mentioned applications, aluminum composites containing Nano WS_2 particles have been used in this study to isolate the results and investigate the effect of the particles and their mechanism of action explicitly by press and sinter technique.

Experimental procedure. The powder metallurgy technique is used with compression testing machine to consolidate the powders. The tribological tests are conducted at room. Microstructural characterization of the powder, composite and post wear samples is performed at every stage using different methods such optical microscope (OM), scanning electron microscope (SEM), and Raman spectroscopy.

Powders. Aluminum metal powder (SRL) 325mesh with 99%purity and air atomized irregular particles was used as the matrix material and control sample. Fig 1 (a, b) show SEM images of the appearance of the as-received aluminum powder at low and high magnification.

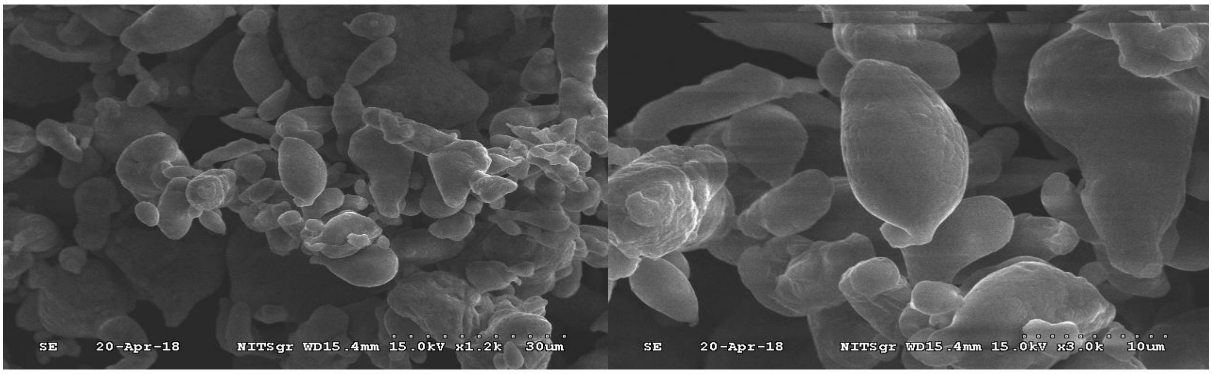


Fig.1 SEM Image of As-received Aluminum Powder at a) Low and b) High Magnification

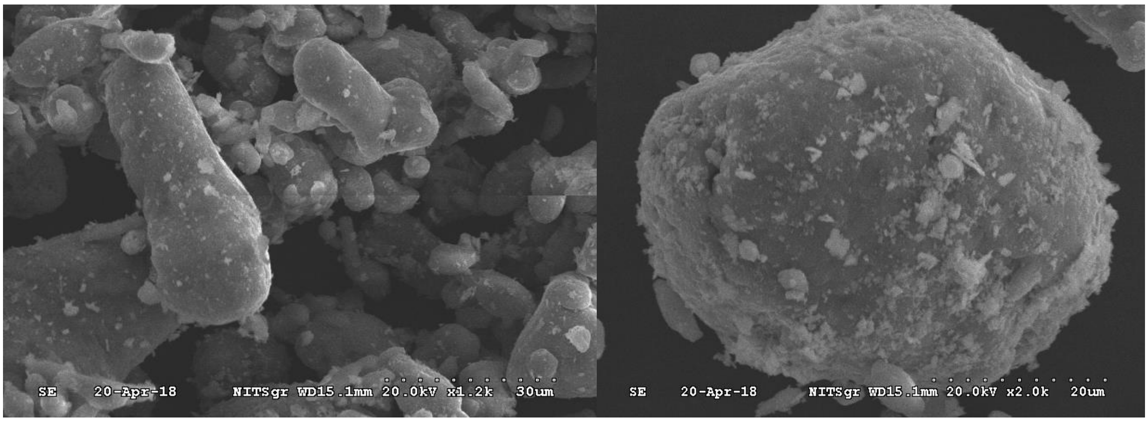
Nano powder of tungsten disulphide was purchased from Nano Shell Company preliminary characterization of WS_2 powder as received is obtained by S.E.M. The Nano powder has 2D flaky structure with an average size of 90 nm and a true density of 7.5 g/cm^3.

MATERIAL AND METHODS. Our objective was to synthesize powders containing 2,4,6 vol.% of WS_2 additives to Al powder. The powders were mixed by the wet chemistry technique. Pure Al powder was added to acetone and ultra-sonicated for 20 minutes using probe. Ultrasonicator. Similarly, WS_2 powder was added to acetone and Ultra sonicated for 20 minutes. The amount of WS_2 and Al powders added were computed to result in 2,4,6 vol.% composites. After sonication of WS_2 the slurry was respectively added to the beaker containing Ultra sonicated Al powder. Al-WS2 slurry was further ultra-sonicated for 20 minutes. The composite mixtures pure Al, Al-2,4,6 % vole WS_2 vol.% were put into oven at 55° for drying. Ultra sonication prevents agglomeration of nano particles thereby improving the homogenous mixing of composites.

(a) (b)

Fig. 2 (a)Probe ultrasonicator and (b) pestle mortar for mixing

The SEM of the mixed powders after mixing and consequent drying at low and high resolution is given in Fig.3.



(a) (b)

Fig. 3 SEM Image mixed Al and WS_2 Powder at a) Low and b) High Magnification

Powder Compaction**.** Compaction is an important step in powder processing as it enables the forming of loose metal powders into required shapes with sufficient strength to withstand till sintering is completed. It's the subsequent sintering operation which imparts final strength. Although there are various methods of powder compaction in the present work a compression testing machine was used to press the compacts to 250MPa.

Before inserting the powder into metallic die, a mixture of 10:1 acetone to triethanamine was added as a binding agent. The binder was mixed to the composite powders in a mortar and pestle for 5 minutes. Further, the die, punch and the stopper was dusted with zinc stearate as a lubricant for easy ejection of compacts. The powder mixtures were then compressed at room temperature for 6 minutes under a pressure of 250 MPa. The compacts were then safely ejected on a universal testing machine. Three sets of samples were prepared. Sintering. Green compacts thus produced were sintered in a high temperature tube furnace at 600°C for one hour in controlled argon atmosphere. Sintering of aluminum is typically carried out at a temperature between 570°C and620°C.Also, Argon was chosen as it provides positive response for sintering aluminum along with nitrogen. The temperature vs time graph for the sintering process is shown in the Fig. 4 The samples were heated at a rate of 5°C / min.

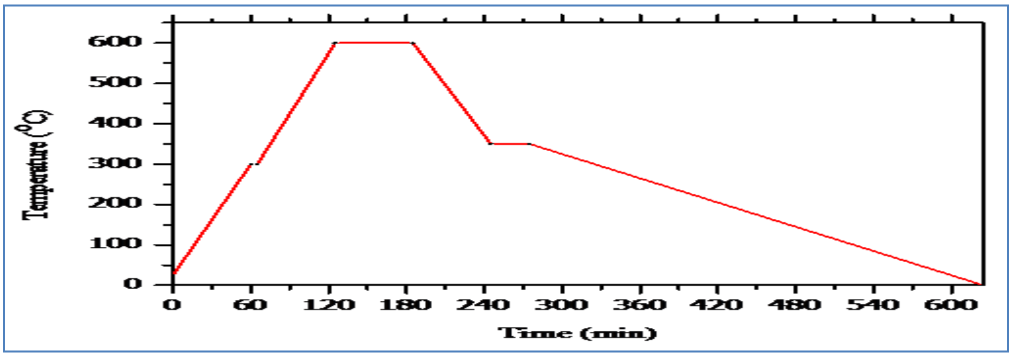


Fig. 4 Temperature vs Time graph for the sintering process

Polishing. The final step in sample preparation is the polishing of the samples for carrying the experiments. The samples were polished with SiC papers of sizes 600,800, 1000, 1200, 1500 and 2000.The final finish was given using alumina powder on velvet paper.

Table 1

Percentage composition of composites

No.	Composites	Composition
1	0	100 % Al
2	I	98% Al + 2% WS_2
3	II	96% Al + 4% WS_2
4	III	94% Al + 6% WS_2

RESULTS AND DISCUSSION. P/M grade aluminum powder has an average size in the range 40–100 μm. Because of its great affinity for oxygen, each aluminum powder particle is essentially an Al-Al_2O_3 composite in dry air. As the Raman characterizes the powders well with peaks below 900 for Al representing its oxides and the peaks of 348 and 414 for WS_2 as shown in Fig.5. Raman spectroscopy was used to characterize the structure of structure WS_2. Density Measurement. The density of green compacts and sintered composite was measured using ASTM B962-17 standards by Archimedes principle. The theoretical density of composite material was calculated by rule of mixtures. Moreover, the calculated relative and theoretical densities were measured by taking mean of three readings per sample. Density Measurement. The density of green compacts and sintered composite was measured using ASTM B962-17 standards by Archimedes principle. The theoretical density of composite material was calculated by rule of mixtures. Moreover, the calculated relative and theoretical densities were measured by taking mean of three readings per sample.

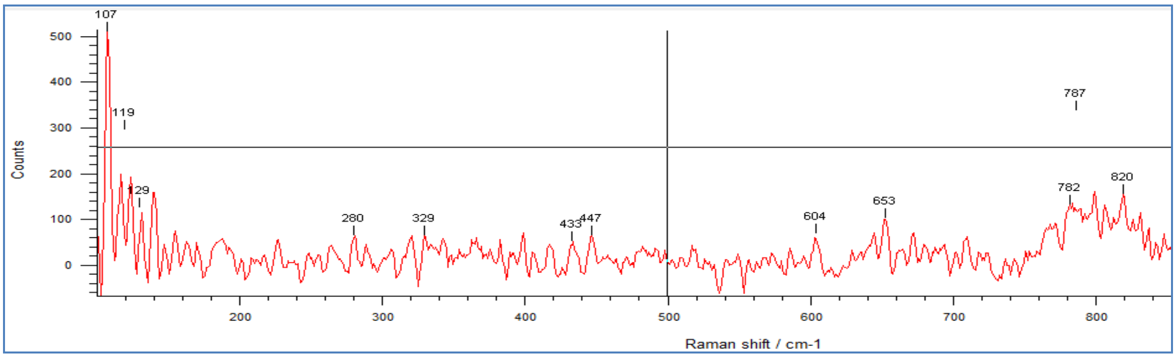


Fig. 5. Raman spectroscopy of Aluminum powder showing its oxides

Density Measurement. The density of green compacts and sintered composite was measured using ASTM B962-17 standards by Archimedes principle. The theoretical density of composite material was calculated by rule of mixtures. Moreover, the calculated relative and theoretical densities were measured by taking mean of three readings per sample. Densities of green and sintered samples are shown in Table 2 and 3 respectively. The variation of densities in the case of green and sintered densities are shown in Fig. 6.

Table 2

Density values of green samples

Samples	Green Density (g/cm^3)	Theoretical Density (g/cm^3)	Relative Density (%age)	Porosity (%age)
0	2.511	2.7	93	7
I	2.636	2.796	94.3	5.7
II	2.621	2.892	90.6	9.4
III	2.842	2.988	93.2	6.8

Table 3

Density values of sintered samples

Samples	Sintered Density(g/cm^3)	Theoretical Density(g/cm^3)	Relative Density(%age)	Porosity(%age)
0	2.6109	2.7	96.7	3.3
I	2.6406	2.796	94.4	5.6
II	2.62554	2.892	90.78	9.22
III	2.73485	2.988	91.5	8.5

The green density of samples is seen decreasing with respect increase on % of WS_2 owing to the reason that its larger particles control the packing and sintering behavior. Since the aluminum powder had irregular morphology and oxidative nature of the powder, it leads to void growth and compact swelling and increased inter granular spacing with WS_2particles over the surface and poor sintered aluminum particles causing agglomerations adds to the decreased density values.

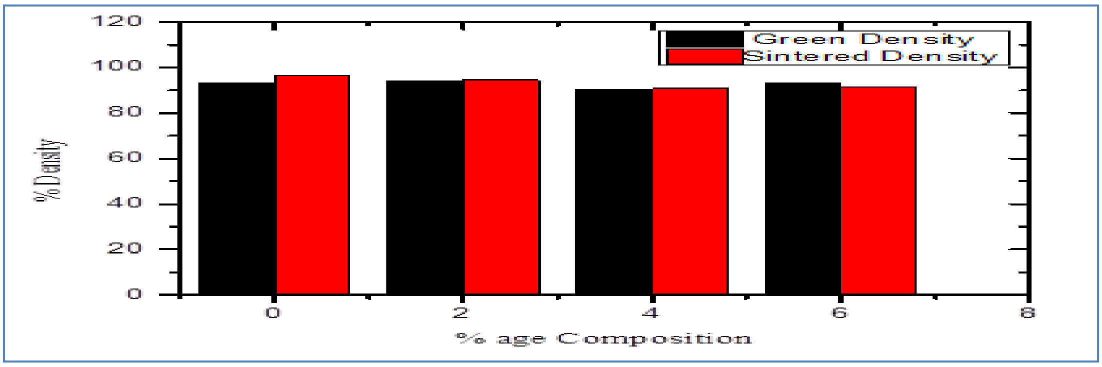


Fig. 6 Green and Sintered density variation of composites

The decreased sintering density is attributed to Kirkendal Effect. Microstructure and Chemical Composition**.** Etching of the samples was carried under ASTM E340 − 15 standards for aluminum i.e., with a solution of 10 g NaOH in 100 mL distilled H2O by Immersing samples for 5 to 15 min in solution heated to 140 to 160°F (60 to 70°C), and then Rinse in water, and dried. SEM images provided information about dispersion of the additives into the matrix and morphology of the WS_2 additive as shown in Fig. 7.

Also, these images allow to identify how the additive interact with Al matrix, giving information about grain size and trapped porosity of the sintered samples. The microstructure confirms the presence of WS_2 powder along the grain boundaries and over the surface without effective fusion of the nano powder into the matrix. Further analysis confirms the presence of oxidative layers over the aluminum grains which are pulled at certain locations giving a porous look of the grains. The grain size was analyzed by metallographic application suite.

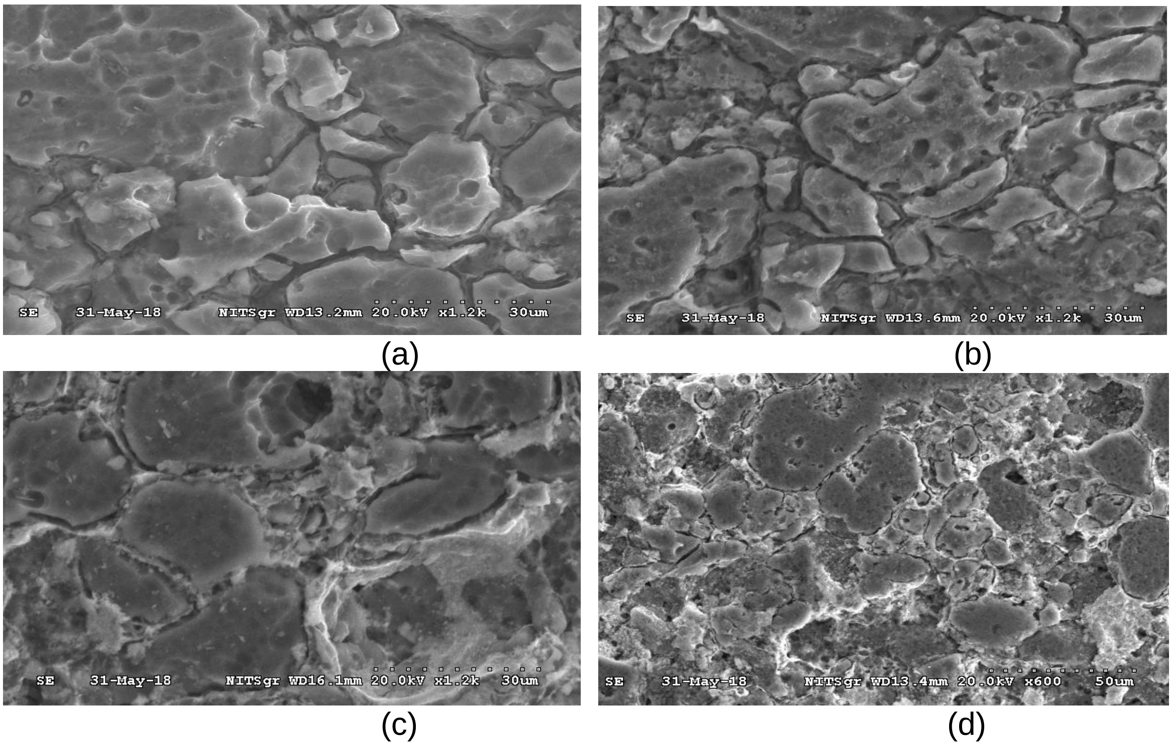


Fig. 7 SEM images of etched samples a) 0 composite, b) I composite c) II composite, d) III composite

A downward trend is seen in the grain size, thereby making an inference smaller the grain size harder the material as shown in Fig. 8. Hardness Test**.** Hardness of a material provides useful information which can be correlated to itstensile strength, wear resistance, ductility, and other physical characteristics. The hardness tests were carried on INNOVA FALCON 500 Hardness Tester, with various loads 100g,200g,3000g,500g with constant dwell time of 10 s as shown in Fig. 9. The samples show a decrease in hardness values with increase in percentage composition. This is attributed to decreased sintered density as porosity damages mechanical properties.

Table 4

Grain size variation with Ws2 percentage

S. No.	Composition	ASTM grain size
1	0	7.47
2	I	7.30
3	II	6.24
4	III	6.19

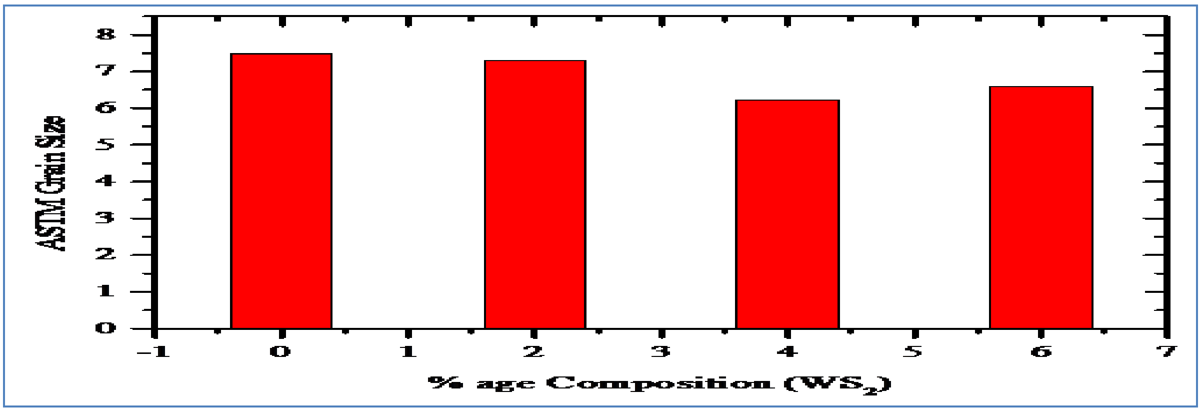


Fig. 8 Grain size variation with Ws2 percentage

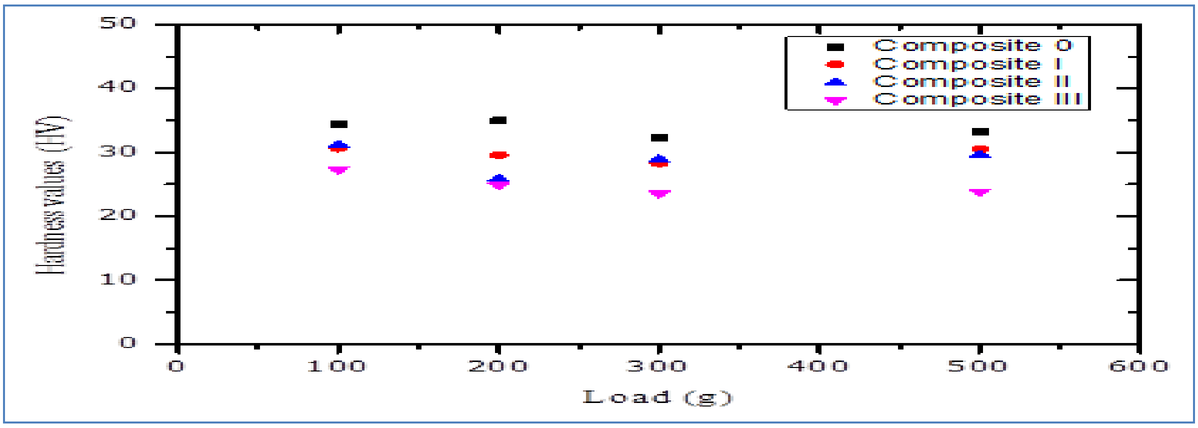


Fig. 9 Variation of hardness with load for different % age composition

CONCLUSION. In this research study aluminum MMC by adding different percentages of WS2 using P/M sintering technique. It is concluded that P/M plays significant role obtaining MMC with different densities and hardness. The rate of heating of sintering process culminates into formation of various oxides. The Strength of the composites increased up to 4% volume and then decreased owing to more agglomerations with the increase in percentage WS_2.With the increase in WS_2 percentage the hardness values showed a decline, the reason being accredited to higher agglomerations of Al-Al and Al-WS_2 particles.

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