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Coating of Synthesized Molybdenum Nanopowder on Aluminium

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ABSTRACT
Surface quality of industrial components is enhanced by molybdenum coating. Molybdenum which has high wear resistance and corrosion resistance is used to reduce friction between the moving parts. An experimental investigation of coating on the aluminium substrate using molybdenum nanoparticles was carried out by spin and dip coating methods. It was found that uniform coating was obtained by dip coating method compared to spin coating. The hardness measurement carried out by the Vickers hardness test showed that this coating increased the hardness of the surface by 1.84 times.

Key words: Spin coating, dip coating, wear resistance, micro hardness, scanning election microscopy, x-ray diffraction

INTRODUCTION
Nanotechnology can be defined as a research for the design, synthesis and manipulation of structure of particles with dimension smaller than 100 nm (Mallikarjuna *et al*., 2012). The importance of research in nanotechnology and the need to acquire intellectual property rights have been emphasized by Bastani and Fernandez (2005). Molybdenum has a beneficial effect on the properties of metallic coatings, improving their hardness, abrasion resistance and corrosion resistance (Chassaing *et al*., 2004; Beltowska-Lehman, 2002; Gomez *et al*., 2005). Earlier investigations were conducted using molybdenum coatings on the AISI 1045 steel substrates using the electro-thermal explosion directional spraying (Jin *et al*., 2007). In this work, an investigation was carried out by coating molybdenum nanoparticles on the aluminium substrate to study the microstructure and the mechanical properties of the coated specimen. The molybdenum nanoparticles were synthesized using planetary ball mill with the sizes ranging from 10-20 nm. TEM images in Fig. 1 show Molybdenum nanoparticles after 30 h of ball milling. The purpose of the nanocoating is to reduce friction and to improve wear resistance between the moving parts in engines. This coating may improve the surface finish, corrosion and heat resistance and also increase the life of the products.

MATERIALS AND METHODS
Spin and dip coating were the coating methods applied. The experimental procedure carried out for coating is as follows:

Spin coating: When flat substrates have to be coated uniformly, spin coating is used (Emslie *et al*., 1958). An excess amount of a solution is placed on the substrate, which is then rotated at high speed in order to spread the fluid by centrifugal force. In spin coating, rotation is

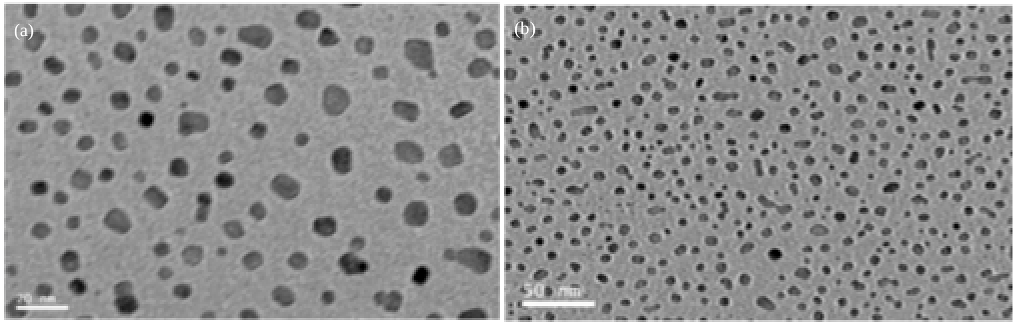

Fig. 1(a-b): TEM images of Mo nanoparticles after 30 h of ball milling

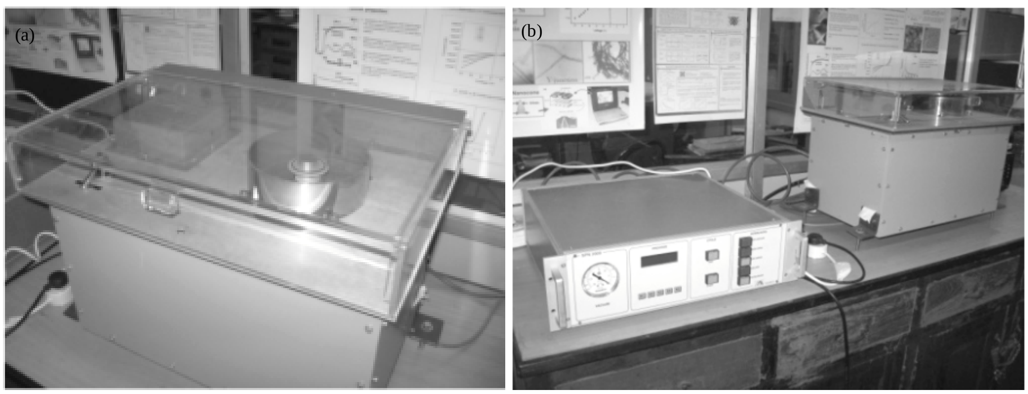

Fig. 2(a-b): Spin coating equipment

continued while the fluid spins off the edges of the substrate, till the required thickness of the coating is achieved. The applied solvent is usually volatile and simultaneously evaporates. By increasing the angular speed of spinning, a thinner film is obtained. The thickness of the film is decided by the concentration of the solution and the solvent. This method is widely used to create thin films with thicknesses below 10 nm. An aluminium plate of the size 30×30×10 and ø30×10 mm was used as a substrate. The surfaces were thoroughly ground by standard emery sheets having numbers of 100, 200, 300 and 400. Then the surface was polished with alumina powder to improve the adhesiveness and substrate bonding strength of coating. The scratches were removed and flat surface was obtained. After polishing the sample was thoroughly cleaned in running water and dried using an air drier. Then the coating was carried out.

The synthesized molybdenum powder was thoroughly mixed with phenolic resin, methyl and di-acetone alcohol solution. The contents were stirred for few minutes to obtain homogeneous mixture. The coating was carried out using MILMAN-SPN 2000 spin coating machine (Fig. 2). The work piece was kept in the spin coater and fastened by means of vacuum chuck. The nanoparticles-resin binder mixture was slowly poured on the work piece for sufficient quantity.

There were two stages in spin coating process. Initially the spin coater rotated at 2000 rpm for 20 sec and then for 10 sec it was stopped. Again spin coater rotated at 4000 rpm for 25 sec and then it was stopped. The work piece was removed from the coater by releasing the vacuum and dried in air for ten minutes. The work piece was cured at 120°C for 1 h in the oven. Then, the microstructure of spin coated portion was analyzed by microscope.

Fig. 3: Dip coated components

Dip coating: Dip coating is a way of depositing a uniform thin film of liquid onto a substrate for solidification into a coating (Scriven, 1988). The coated piece can then be dried by force-drying or baking. It is used to create thin film coated materials.

The synthesized molybdenum powder was thoroughly mixed with phenolic resin, methyl and di-acetone alcohol solution. The contents were stirred for few min to obtain homogeneous mixture. The work piece was kept in a fixture and the powder-resin binder mixture was kept in a container.

The sample was slowly dipped in the mixture for few seconds and taken away from the container. The dipped portion was dried in air initially for 10 min and cured in an oven at 120°C for one hour. The dip coated components are shown in the Fig. 3. The coating adhesion with the substrate was checked by tape test and it was observed that a good adhesion took place. The microstructure of coated portion was analyzed by SEM. The XRD test was carried out to find if any phase change in crystal structure after coating. Vickers micro hardness test was carried out for coated and uncoated portions to check the hardness.

RESULTS AND DISCUSSION

Microstructure of spin coated surface: The spin coated surface was analyzed by LEICA DMLM metallurgical microscope and the images are shown in Fig. 4. The image shows that the molybdenum nanoparticles were not spread uniformly on the substrate material. In most of the places substrate was not coated which are clearly visualize from the images.

Microstructure of dip coated surface: The scanning electron microscopy (SEM) study was carried out using FEI-QUANTA 200 make equipment. The SEM images of dip coated surface are shown in Fig. 5. The coating is uniform on the substrate material surface. It can be seen from the SEM images that some agglomerated nanoparticles are present on the coated surface. From the images it is concluded that the substrate coated using the synthesized molybdenum nanoparticles by dip coating method was good compared to spin coating method.

XRD study of dip coated surface: Figure 6 shows that the x-ray diffraction (XRD) pattern of synthesized molybdenum nanoparticles after 30 h of ball milling which matches with standard PCPDF # 895023, which represents Mo particles with BCC structure. XRD study for dip coated surface was carried out using D8-DISCOVER X-ray diffractrometer. There are totally 9 numbers of peaks obtained. In which 4 nos. of peaks 2θ values in x-ray diffractogram are identical with

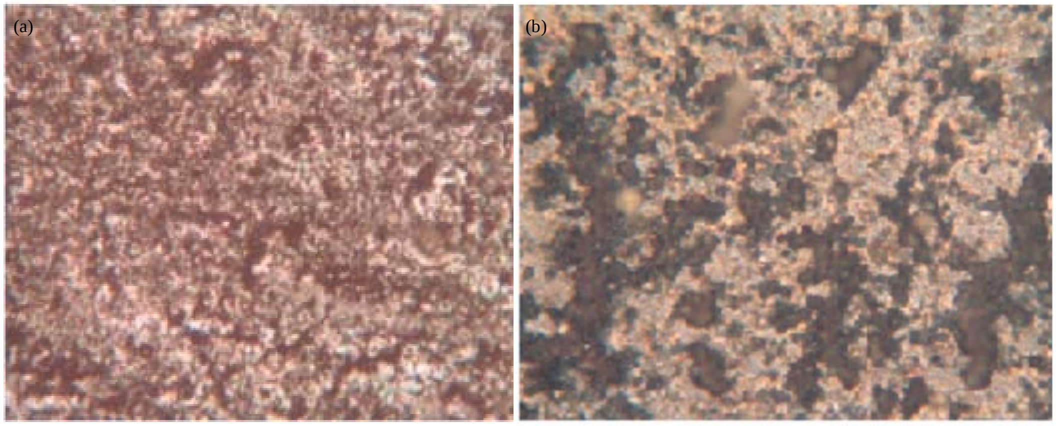

Fig. 4(a-b): Spin coated surface microscopy images, magnification at(a)10X and (b) 50X)

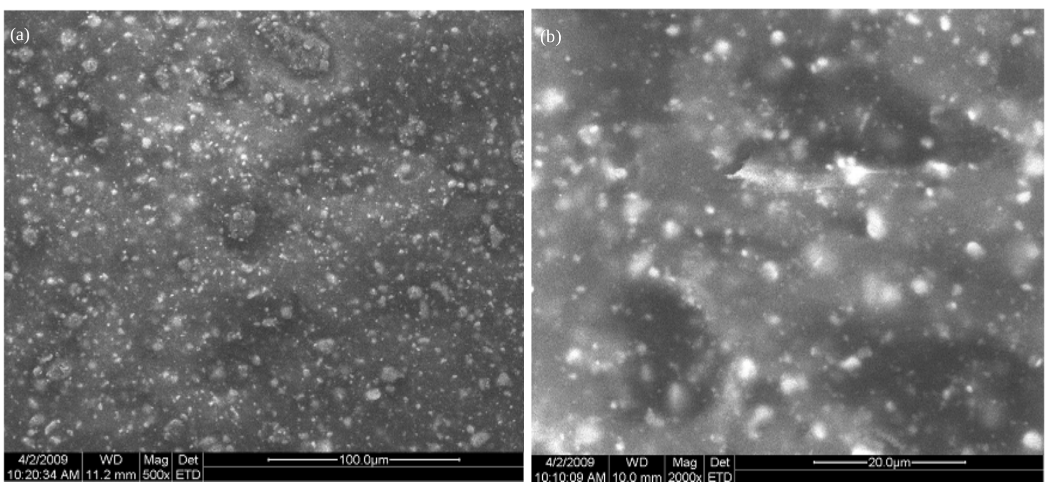

Fig. 5(a-b): Dip coated surface SEM images

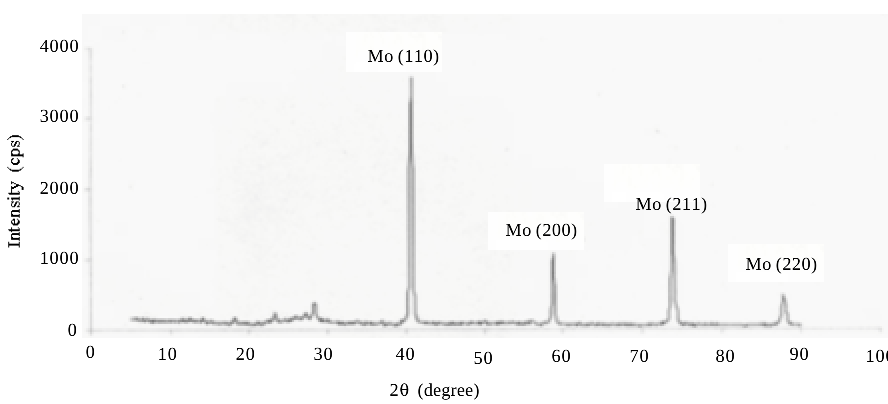

Fig. 6: XRD pattern of synthesized Mo nanoparticles

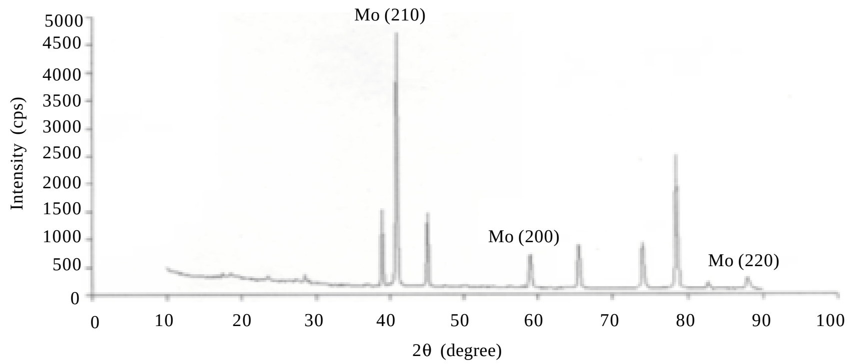

Fig. 7: XRD pattern of synthesized Mo nanoparticles

Table 1: Vickers hardness at uncoated portion

D_1 (μm)	D_2 (μm)	Hardness (VHN)
61.9	61.8	49.0
60.1	60.0	51.5
65.9	65.9	42.7
64.6	64.6	44.4
63.6	63.5	45.9

Table 2: Vickers hardness at Mo nanoparticle coated portion

D_1 (μm)	D_2 (μm)	Hardness (VHN)
45.2	45.2	88.5
47.9	47.9	80.8
44.7	44.5	93.6
47.9	47.8	80.8
46.7	46.7	85.0

PCPDF # 895023, which represents Mo particles with body centered cubic (BCC) structure with reference to Fig. 7. Remaining 5 nos. of peaks 2θ values are identical with PCPDF # 882331 which represents Mo particles with face-centered cubic (FCC) structure with reference to Fig. 7. From this, it is concluded that the coating consists of both BCC and FCC structured Mo particles. Some of the Mo particles phase was changed from BCC to FCC after coating and curing process. The curing at 120°C for an hour is the reason for phase transformation.

Hardness test: The hardness measurement was carried out by the Vickers hardness test using MMT-7 Matsuzawa Micro hardness tester, Japanese make. The load applied for hardness measurement was 100 gf. The hardness test was conducted on both the uncoated surface of the substrate and molybdenum nanoparticle coated surface (Table 1, 2).

With reference to the above tables the average value of Vickers hardness for uncoated surface is 46.7 VHN and for coated surface is 85.74 VHN. Mo nanoparticle coated surface hardness increased about 83.6% compared to the uncoated surface. This means that the coated portion is having about 1.84 times higher hardness than the uncoated portion.

CONCLUSION

The molybdenum nanoparticles synthesized by planetary ball mill with the sizes ranging from 10-20 nm were coated on the substrate by Spin coating and Dip coating methods. It was found that uniform coating was obtained on substrate surface by dip coating method. The dip coated portion was analyzed by SEM. From XRD analysis, it is observed that phase transformation in molybdenum crystal structure occurred from BCC to FCC and both were present at coated portion. The hardness measurement test using Vickers hardness test method was carried out and it is found that the coated portion is having about 1.84 times higher hardness compared to uncoated portion.

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