

Sized WS₂ Lamellae and their Texture Orientations Growth

KHALIL EL-HAMI¹, CRISTINA LOURO² and ALBANO CAVALEIRO²

¹Univ. Hassan 1, Faculty of Khouribga, Laboratory of Nanosciences and Modeling,
PB:145,Khouribga City, Morocco.

²Univ. Coimbra, SEG-CEMUC, Center of Mechanical Engineering,
Polo II, 3030-788, Coimbra city, Portugal.

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ABSTRACT

The tungsten disulfide (WS₂) thin films, considered as inert, non-toxic, non-corrosive lubrication that is resistant to most fuel solvents, is the best solid lubricant for general industry. In this paper, WS₂ thin films were deposited on steel and silicon <110> type substrates by DC sputtering methods with optimized experimental conditions and parameters. Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) were employed to characterize the surface morphology and determine the geometrical size of WS₂ lamellae or microtubes. The investigation showed that both SEM and AFM analysis revealed oriented WS₂ crystalline lamellae on silicon substrate while they are entangled on the steel substrate. This result leads to say precociously that the WS₂ thin films deposition depend on the substrate type. Moreover, using the AFM results, we could determine the average lamella (or microtube) size which has from 200 nm to 500 nm in width and from 1mm to 1.5mm in length. The EDS tool allows attaining the composition of deposited WS₂ where the tungsten and the disulfide represent about 40 and 55%, respectively.

Key words: Tungsten disulfide, Scanning Electron Microscopy, Atomic Force Microscopy, EDS, WS₂ lamellae, texture orientations.

INTRODUCTION

The application of thin films for tribological and mechanical devices has attracted considerable interest in the recent past¹ Especially; the tungsten disulfide (WS₂) has been largely studied and characterized for various applications²⁻⁸.

The main application of the WS₂ is used for solid film lubricant. Physical vapor deposition (PVD) is a widely used technique of depositing thin films by sputtering, that is ejecting, material from a target, that is source, which then deposits onto a substrate⁹. Sputtered atoms or ions ejected from the target have a wide energy distribution, at higher gas pressures, they collide with the gas atoms that act as a moderator and move diffusively, reaching the substrates or vacuum chamber wall and condensing after undergoing a random walk. The sputtering gas is often an inert gas such as argon. Sputtering is used extensively in the semiconductor industry to

deposit thin films of various materials in integrated circuit processing. Thin antireflection coatings on glass for optical applications are also deposited by sputtering¹⁰.

EXPERIMENTAL

Tungsten disulfide WS₂ thin films were deposited on steel and silicon <110> type substrates by DC sputtering methods. We chose rotating than fixed substrates, in the sputtering chamber, in order to reduce the coefficient of friction and wear and obtain a long wear life of the film. On the other hand, rotating substrates have economic impact by using lot of samples. The efficiency of the solid film lubricant depends on the degree of adherence of the film to the surface to be lubricated. For this reason, the surface pretreatment with argon in ultra-high vacuum, the energetic of the sputtered material using approximately 700 V, the type of the substrate materials and type of interface formed play a key

role for the adherence process and determines the durability and wear of the sliding components.

RESULTS AND DISCUSSION

Optical microscope characterization

From optical microscope image (Fig.1) we can see a large area of the deposited WS_2 film showing homogenous and uniform film without holes or roughness in Fig.(a), and the standing WS_2 lamellae as rod-like in Fig.(b). This result was due

to many experiments on DC sputtering methods to optimize all parameters and conditions such as value and time of applying voltage, argon ratio, etching condition, distance between target and substrate and so on.

Profile-meter determination

To determine the thickness of the deposited WS_2 film, we used a profile-meter technique. A step, suitable for surface profiling, was provoked on the film edge by using boron nitride and then

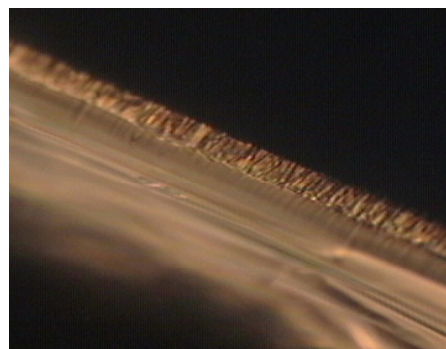


Fig.1: Optical microscope image of WS_2 on Si substrate, (a) on the top, (b) at the edge (100x100mm)

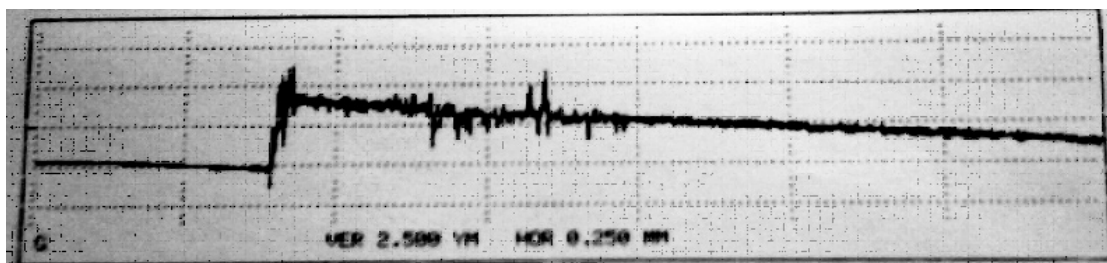


Fig.2: Profile-meter of WS_2 film

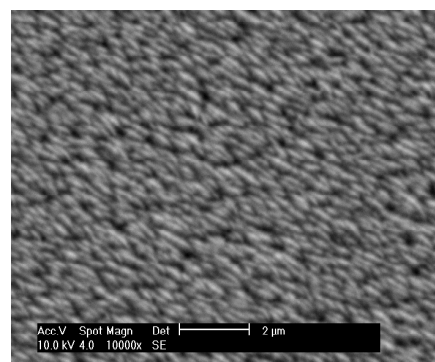
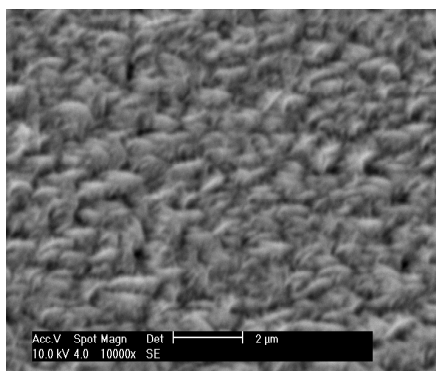


Fig. 3: SEM morphology image of WS_2 on (a) Steel and (b) on Si

acetone. The detection of the scanned edge gives the projected wall thickness. The profile-meter indicates a WS_2 film thickness of about 2.5 nm as shown in Fig.2. We notice the presence of significant drift.

SEM characterization:

The SEM images in Fig. 3 reveal oriented WS_2 crystalline lamellae on Si substrate than on the steel substrate. At first study, we can suggest that the structure of WS_2 lamellae took a preferential

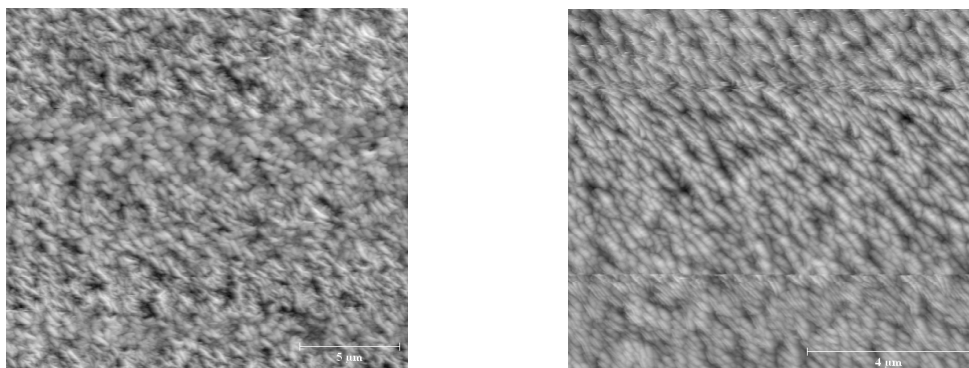


Fig.4: AFM topography image of WS_2 on Steel (a) Steel and (b) on Si

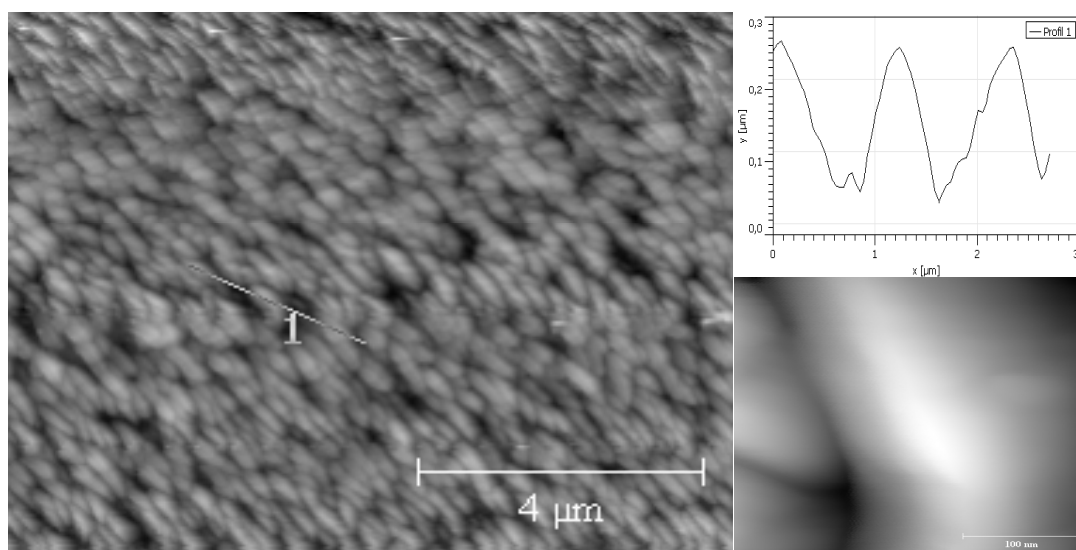


Fig.5: AFM image of 10 mm scanned area (a), AFM line profile (b), AFM image of individual lamella (c)

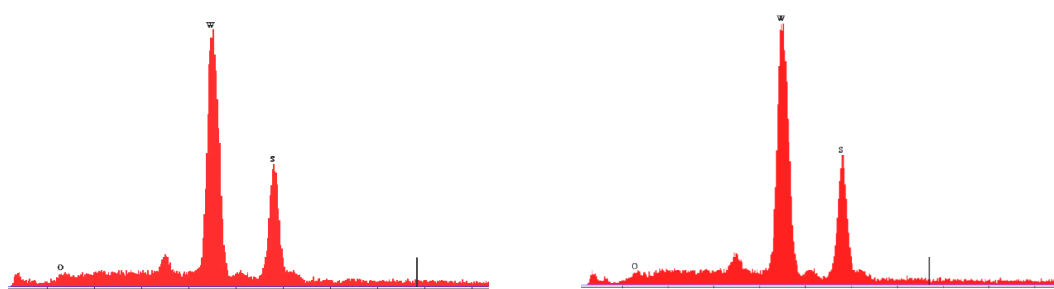


Fig. 6: EDS spectrum of WS_2 on steel (a) and on Si (b)

orientation on Si <110> substrate while they were entangled on the steel substrate.

AFM characterization

The tapping mode AFM is used to obtain topography images of WS₂ thin film on both steel and Si substrates. The conditions of the tapping mode used in this investigation with a tip of silicon doped under the frequency of 373 KHz, stiffness of 30 N/m and a speed of 0.5 Hz.

The AFM topography images showed the same observation as in SEM in which WS₂ crystalline lamellae are oriented on Si substrate in a preferred direction than on steel substrate. We notice that we used a Si wafer <110> and the WS₂ lamellae are following these directions. The suggestion may lead to say precociously that the film deposition depends on the substrate.

Referring to the Fig.5, the AFM image shows homogeneous and uniform thin film of WS₂ containing crystalline lamellae texture. The geometry of individual lamella is sized from 200 nm to 500 nm in width and from 1mm to 1.5mm in length.

EDS characterization

The characterization of WS₂ on steel

substrate by EDS is shown in the spectrum of the Fig. 6. As we can see, the tungsten (W) and sulfide (S) represent, respectively, ratio of 40,83% and 53,39%. The approximately ratios of W and S were found on the Si substrate (Wratio = 41,04% and Sratio = 53,15%).

CONCLUSIONS

The investigation on sputtered tungsten disulfide (WS₂) thin films showed different textures of crystalline lamellae deposited on steel and silicon substrates. These preferred orientations influence and will have effect on tribological properties of WS₂. Further experimentations concerning the growth of WS₂ on different surfaces like KBr, NaCl or highly ordered pyrolytic graphite (HOPG) substrates could be interesting and will be a widespread topic of the coming investigations

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