Serbian Tribology Society

SERBIATRIB '09

11th International Conference on Tribology

University of Belgrade Faculty of Mechanical Engineering

Belgrade, Serbia, 13 - 15 May 2009

CHARACTERIZATION OF NANO CRYSTALLINE LUBRICANT COATING TO STUDY THE EFFECT OF GRAIN SIZE AND PHASE STRUCTURE ON THE COATING BEHAVIOR

Norm Gitis[1] ,Vishal Khosla[1], Paolo Bariani[2]
[1]CETR, Inc., 1715 Dell Avenue, Campbell, CA 95008, www.cetr.com
[2]Schaefer South-East Europe, via Quirina 1313, I-45031 Rovigo, Italy

Abstract: *This paper presents the techniques used to comprehensively characterize the effect of current density on the grain size, phase structure and the corresponding effect on the tribological, corrosion, wear and adhesion behavior of nanocrystalline lubricant coating.*

Keywords: *Nanotribology, lubricant, Nano Crystalline coating*

1. INTRODUCTION

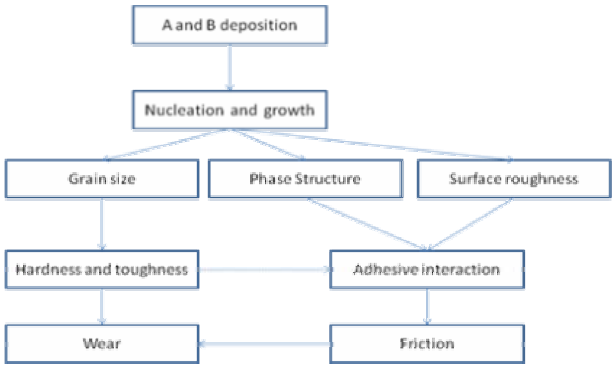

Figure 1. Schematic showing effect of pulsed deposition on final coating structure

Nanocrystalline materials, with smaller grain size (order of some 100nm) used for lubrication and wear resistance, have been the subject of intensive research. Among many processing techniques developed for producing nanocrystalline materials pulse electrode deposition has received considerable attention in recent years, owing to the possibility of changing the final coating properties by regulation of pulse parameters. [1] This paper investigates the techniques used to comprehensively characterize the effect of depositing conditions on the tribological, corrosion, wear and adhesion behavior of nanocrystalline coating.

Figure 2. UMT picture

The figure 1 summarizes the deposition of a coating from two precursors A and B. It is imperative to understand the grain size, phase structure, surface roughness, hardness, etc to predict the wear and frictional properties of the coating. Traditionally each characterization step requires a separate instrument (nanoindenter, microindenter, AFM, adhesion tester, tribometer etc.). This study evaluates and shows the capabilities of a single tool, model UMT to

comprehensively characterize all the desired properties. Figure 2 shows the UMT picture which was used for this study.

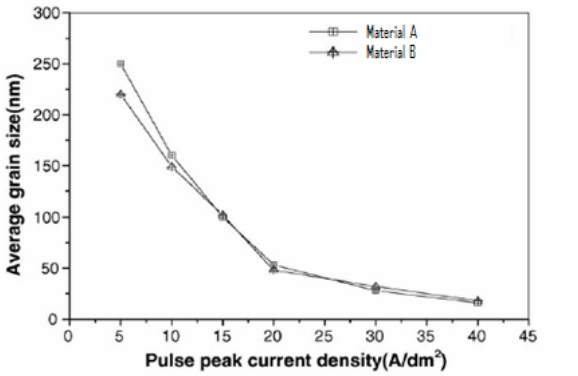

Figure 3. Grain Size of coatings deposited with different current density

Nanocrystalline material A and B coatings with different grain sizes were fabricated through the control of peak current density. The average grain size of the coating was calculated using the integrated AFM on UMT . Fig. 3 shows the influence of the pulse peak current density on the average grain size of nanocrystalline A and B coatings while keeping other parameters constant. As shown in figure 3 the average grain size of A and B coatings decreases with increasing peak current density. The corresponding SEM images are shown in figure 4. SEM images show that the polycrystalline material A has a rather regular surface and consists of pyramidal and polyhedral crystals. However, the polycrystalline material B showed a rather regularly branched structure.

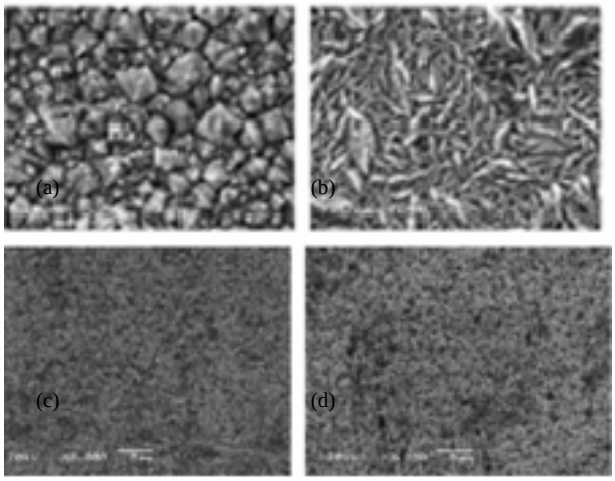

Figure 4. (a), (b) SEM pictures of material A (c),(d) material B deposited at different current density

To measure the effect of depositing parameter on hardness, an integrated instrumented micro – indenter MH-2 was used. The figure 5 shows the change in Vicker's hardness with changing grain size. To estimate the coating uniformity, 10 indentations were done at different positions. The head can also be used to generate hardness or modulus vs depth data. Generally speaking, if the coating is much thinner, the interchangeable nano-head (NH-3) on the same tool can be used to measure hardness, modulus etc vs. depth. [2] Careful consideration should be made to choose the technique and to keep in mind the artifacts involved with each technique. In the shows data it can be observed that the hardness of both nanocrystalline A and B coatings increased considerably with decreasing grain size from micrometer to nanometer, and the slope is not constant but decreases with the increase of grain size.

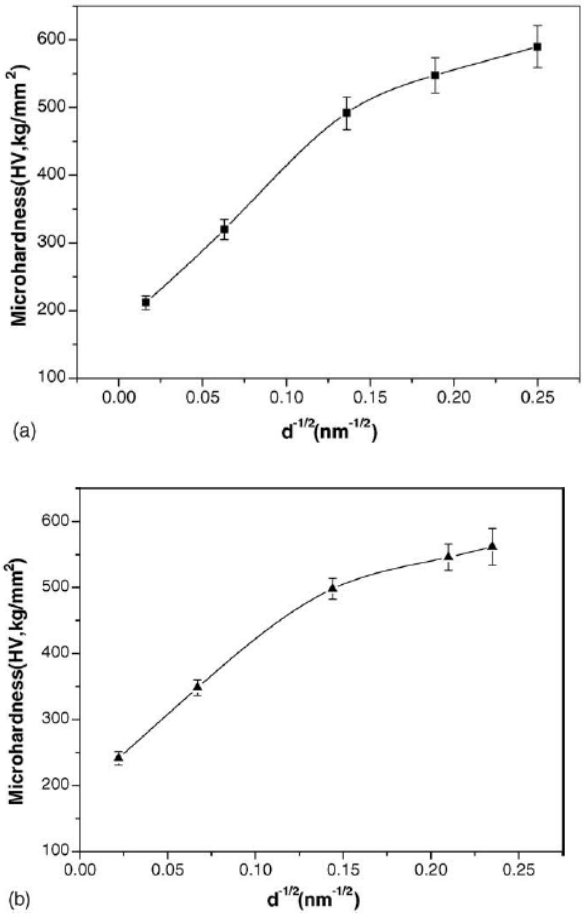

Figure 5. Instrumented Vickers Micro hardness vs. grain size diameter (a) material A, (b) material B

The adhesion of the coating to the substrate can be investigated in pull or peel or shear adhesion mode. For pull-up adhesion, the most important parameter is the holding time before the lift up. The shear adhesion is measuring static friction of the onset of the motion; usually this static friction can be orders of magnitude higher than the dynamic coefficient of friction. Static friction can be measured in rotary, reciprocating or block-on-ring mode (choice should be dictated by the final application of the lube/coating). The peel up mode is only good for thick coatings, where the coating is peeled from the surface at different angles.

The wear and frictional properties of coating can be measured simultaneously on the UMT (rotary,

reciprocating or block-on-ring mode). At all times the tool uses multi sensing technology to measure in-situ friction, wear, temperature, acoustic emission, velocities, etc.[3] For the presented data, friction was measured using a 4 mm stainless steel ball and rotating disk coated sample. Figure 6 shows the wear rate vs micro hardness data. Figure 7 shows the coefficient of friction for both the materials.

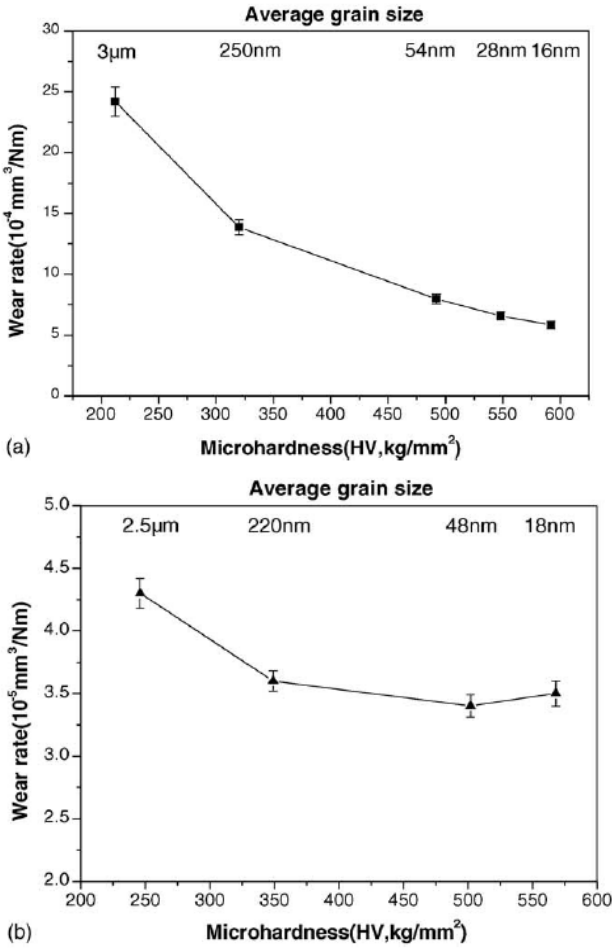

Figure 6. Wear rate vs. Micro hardness (a) Material A, (b) Material B

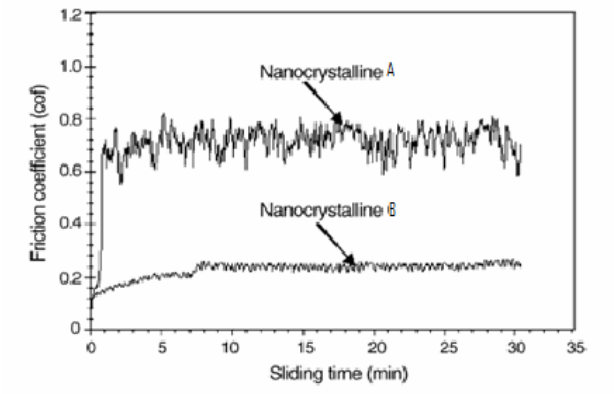

Figure 7. COF vs sliding time

Figure 8 shows the friction coefficient of the coatings with different grain size. The data shows that the frictoin coefficient of material A is twice than that of material B. In addition, the friction coefficients of B coatings were much more stable than that of A coatings, showing lower vibration of friction coefficient. Its also intersting to note that for different materials (and B) the coefficient of friction (COF) changes in a different manner with changing grain size. For material B cof increases with grain size as compared to material A where cof decreases with increase in grain size.

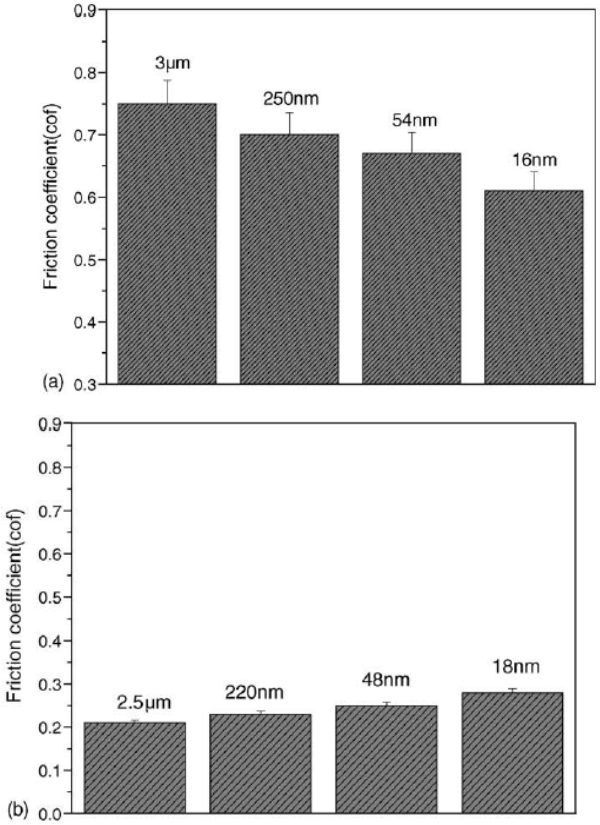

Figure 8. COF vs different grain size (a) Material A, (b) material B

After the test is finished, the wear track can be analyzed under an integrated high-magnification microscope and an AFM (or 3D Profiler). Figure 9 shows the AFM image of the wear track. Figure 10 shows the wear track analysed using a 3 D profiler.

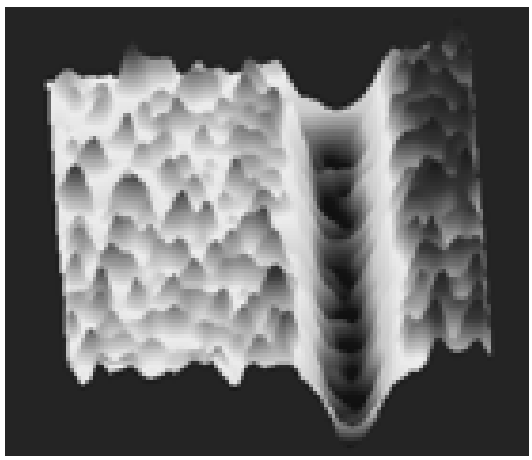

Figure 9. AFM Scan of the wear track

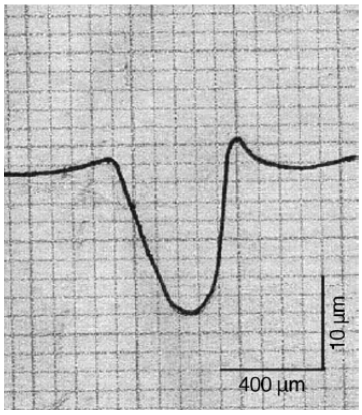

Figure 10. 3D profiler Scan of the wear track

Corresponding SEM images of the wear track are shown in figure 11 and 12.

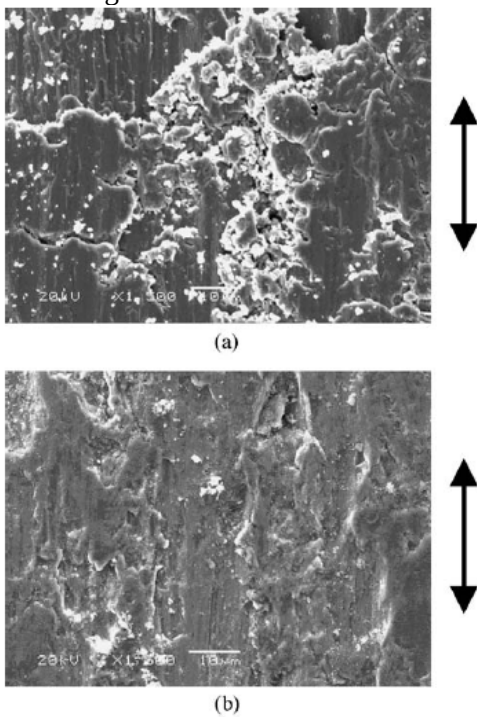

Figure 11. Worn surface morphology of material A

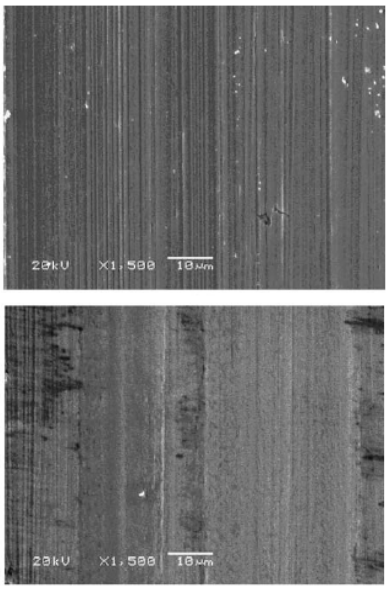

Figure 12. Worn surface morphology of material B

2. CONCLUSION

It is important to perform multiple tests to understand the effect of grain size, depositing conditions etc. on the properties of the nanocrystalline coatings. The Universal Material Tester mod. UMT is effective for such comprehensive studies. It can be used to measure many parameters such as hardness, friction, adhesion, surface roughness, profiles, wear rate etc.

REFERENCES

[1] Liping Wang, Yan Gao, Qunji Xue, Huiwen Liu, Tao Xu Structure properties of novel lubrication and wear resistance gradient (LGM) Ni-Co nano crystalline alloys. 2006

[2] N. Gitis, J. Xiao, and M. Vinogradov, J. ASTM Int. STP 1463, 2 (9), 80(2005)

[3] N. Gitis, I. Hermann, S. Kuiry, V. Khosla, Proc. STLE/ASME Int. Joint Trib. Conf