

## MICROSTRUCTURE AND MECHANICAL AND TRIBOLOGICAL PROPERTIES OF A LUBRICANT COATING FOR INCREMENTAL FORMING OF A TI SHEET\*

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**Abstract**– Due to galling with other materials, Ti and its alloys call for solid lubricants during forming. Deforming Ti sheet becomes even more difficult when the forming process involves very low contact area, such as in the novel process single point incremental forming (SPIF). Retaining lubricant at the tool/sheet interface while performing local deformation, as in SPIF, has always been a challenge. One way to satisfy this challenge is to develop a porous surface coating on the sheet and fill its pores with lubricant particles. For SPIF, an electro-chemical deposition process called micro-arc oxidation (MAO) has shown promise to fabricate coatings of desired pore size and thickness to fulfill the said objective. In the present work, the characterization of an MAO coating has been carried out to clarify its role during the incremental forming of pure Ti sheet. The results regarding hardness, bond strength, XRD microstructure and friction co-efficient of the coating under various lubrication conditions are reported.

**Keywords**– Surface coating, characterization, incremental forming, pure titanium

### 1. INTRODUCTION

Single point incremental forming (SPIF) is a novel sheet metal forming process. It employs a hemispherical-end steel rod (like a mill tool) to impose deformation on the sheet (see Fig. 1), and is characterized by very high localized contact pressure at the tool/sheet interface. The process is able to manufacture parts by employing various sheet metals such as aluminum [1-6], steel [7], copper [8], Ti [9-11] and PVC [12]. The ordinary lubricants such as mineral oil and grease, etc. adequately reduce friction under mild loading; however, these do not serve the purpose when the involved loads are severe [13, 14]. Thus, the oils and greases serve well during cold forming of aluminum and steel. However, during cold forming of Ti these ordinary lubricants are not appropriate due to local and high contact pressure at the tool/sheet interface. The authors in their early works [9, 10] have demonstrated that only solid lubricant, i.e. a paste of MoS<sub>2</sub> (particle size varied from 6 μm to 9μm) and grease (EP-2 Shell make), can withstand the high contact pressure involved in the localized deformation of SPIF. In order to retain the solid-lubricant-particles at the tool/sheet interface, preparation of a porous coating (whose micrograph is shown in Fig. 2) on sheet substrate was also emphasized.

Furthermore, it was suggested that micro arc-oxidation method can be used to obtain the deposition of porous coating with required thickness (i.e. 18.2-30μm) and pore size (i.e. 5.4-9.3μm) on Pure Ti substrate with the flow curve shown in Fig. 3. However, there are a number of issues which need attention before adopting this method on commercial scale. These are as follows:

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\*Received by the editors January 22, 2013; Accepted June 3, 2014.

- (i) Whether the coating can withstand the localized forces due to the mechanics involved in SPIF process and will remain intact or not?
- (ii) Effect of coating on the substrate and vice versa
- (iii) Effect of coating on the quality of formed parts
- (iv) Whether the presence of coating retards/facilitates the process or not?

In this short paper, characterization (mechanical, microstructural and tribological) of porous coating developed previously on pure Ti substrate is carried out to investigate the above-mentioned aspects.

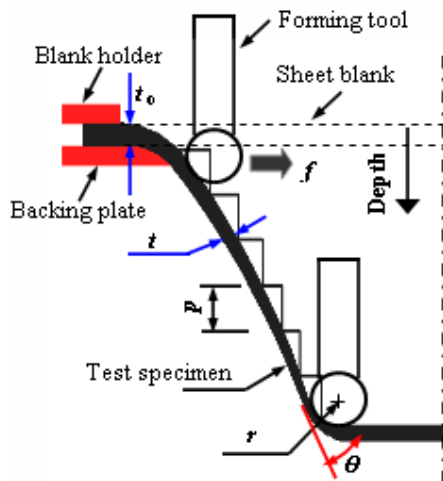


Fig. 1. Schematic and terminology of SPIF process:  $t_0$  is blank thickness,  $p$  is step size,  $r$  is tool radius,  $f$  is feed rate and  $\theta$  is wall angle

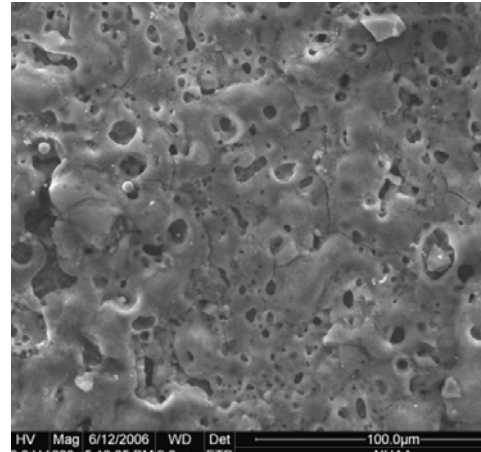


Fig. 2. The surface coating employed for SPIF of Pure Ti [9]

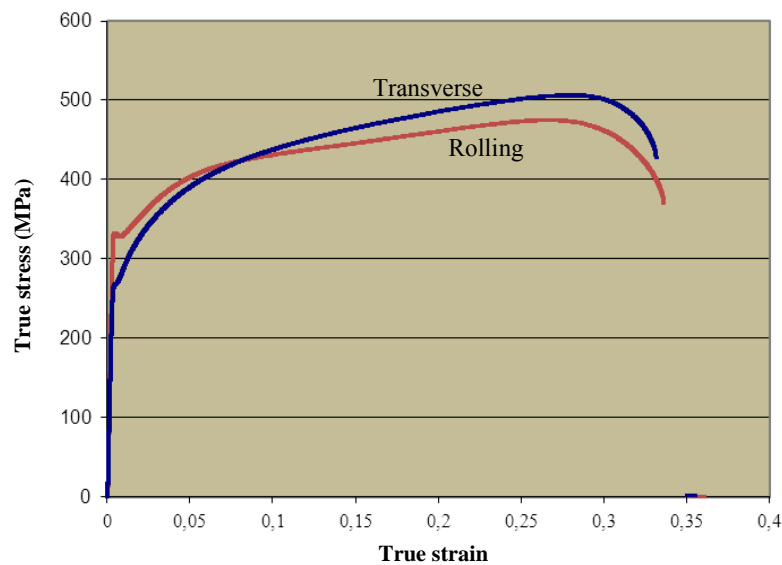


Fig. 3. Flow curve of pure Ti sheet in the rolling and transverse directions

## 2. EXPERIMENTAL DETAILS

Keeping in view the micro-arc oxidation method and particular nature and the mechanics of SPIF process, the following characterization tests have been employed to address the issues highlighted in the preceding section. Each forthcoming test was repeated 3 times and the average results are reported.

### a) Mechanical characterization

The following two tests were conducted in order to mechanically characterize the film: To test hardness across coating thickness, Shimadzu dynamic ultra-micro-hardness system (DUH-W201/W201S) was employed. A Berkovich diamond tip (triangular pyramid) was used as an indenter. The test was conducted as detailed in [15] and under the following conditions:

*Force range = 1000mN, Maximum test depth = 9 $\mu$ m, Holding time = 10 sec, Loading speed = 5mm/sec, and Tip angle of the indenter = 100 $^{\circ}$ .*

The dynamic ultra-micro hardness, also called dynamic nano-hardness, was computed using the formula derived in Uzuna et al. [15] and presented below:

$$H = P_{\max} / A_c = P_{\max} / 26.43h_c^2 \quad (1)$$

where  $H$ ,  $P_{\max}$ ,  $A_c$  and  $h_c$  stand for dynamic ultra-micro hardness, peak indentation force, contact area and contact depth, respectively. To compare the hardness of coating with that of substrate, indentation test was conducted on the Pure Ti sheet (without coating) as well.

The bond strength between the coating and substrate was evaluated by employing WS-97 scratching machine. The test was performed using the following parameters: Scratching velocity = 2mm/min, Scratch length = 6mm, Loading velocity of force = 10N/m, and Maximum set force = 25N.

### b) Microstructural characterization

The microstructure of the coating was determined with Rigaku Y-Q4 X-ray diffraction (XRD) apparatus using a CuK $\alpha$  radiation at a scanning speed of 6 $^{\circ}$ /min. However, prior to scanning, the film was cleaned with acetone. The coating thickness and pore size have been already reported in the previous work [9]; therefore, tests regarding these aspects of coating were not carried out.

### c) Tribological characterization (Sliding friction)

Minutolo and co-workers [16] have employed pin on disk test to determine sliding friction between the incremental forming tool and sheet. The friction co-efficient obtained from this test was used in FE analysis. The FE results were found in good agreement with the experimental ones, showing that pin on disk test can be used for testing friction at the tool/sheet interface in the SPIF process. More details on this specific test have been reported in [16]. In the current study, sliding friction tests were conducted at room temperature using HT-500 pin-on-disk tribometer. The samples under the load of 180g were rotated for 10min at the speed of 260rev/min. The GCr15 balls with 3mm radius and 77HRC average hardness were used as counter-face body. The diameter of wear track was set to 8mm. The sliding speed in this way, based on RPM and wear track diameter, was kept at 6000mm/min, which is comparable with the sliding speed of forming tool used during incremental forming. It is noteworthy to mention that the SPIF tests in [9], a former study by the authors, were performed at the maximum sliding speed of 1500mm/min. However, later on in [10], this speed was increased from 1500mm/min to 6000mm/min. The surface condition was found equally smooth. The friction tests were performed both for the substrate and coating. For coating, the tests were carried out under dry as well as various lubrication conditions.

## 3. RESULTS AND DISCUSSION

### a) Dynamic ultra-micro hardness

Figure 4 presents the ultra-micro hardness of coating and substrate. During the tests it was observed that the hardness was dependent on the position of indenter with respect to the position of pores which were

scattered throughout the surface of the coating. The hardness of coating was found to be the minimum in and around the immediate vicinity of the pores; whereas, it was maximum when the indenter was positioned away from the pore location. It can be seen from Fig. 4 that the coating hardness reduces from a significantly large value of 3GPa to 0.5GPa as the indentation depth from surface increases to 0.8 $\mu$ m. Here-after, the hardness contrarily begins to increase steadily and keeps this trend till 6.5 $\mu$ m where it achieves the maximum value of 0.86GPa. Thus the region between 0.8 $\mu$ m and 6.5 $\mu$ m depth seems to be relatively compact. From 6.5 $\mu$ m to 7.25 $\mu$ m, the hardness again falls down to achieve a stable value of 0.72GPa. The maximum coating hardness in the compact region is about 1.15 times larger than that of substrate (0.75GPa, average hardness measured after removing oxides layer). However, the coating hardness corresponding to the maximum indentation depth (i.e., 8.5 $\mu$ m) is slightly less than that of substrate. These variations in hardness, as evident from the micrograph presented in Fig. 2 and reported in the literature [17], result from the micro-porous nature of MAO coating.

### b) Bond strength

Figure 5 depicts the result of a scratch test conducted to determine the bonding strength between the coating and substrate. The force corresponding to the first sharp peak was regarded as the bond strength. With 3 scratch tests, the average bonding strength was found to be 12N. This indicates that the bond between the coating and substrate is not so strong. However, no cracks were observed in the coating while examining on-job performance of the coating. Therefore, it can be said that the coating under study with the strength of 12N can withstand localized forces of SPIF, and can be successfully employed for the intended purpose.

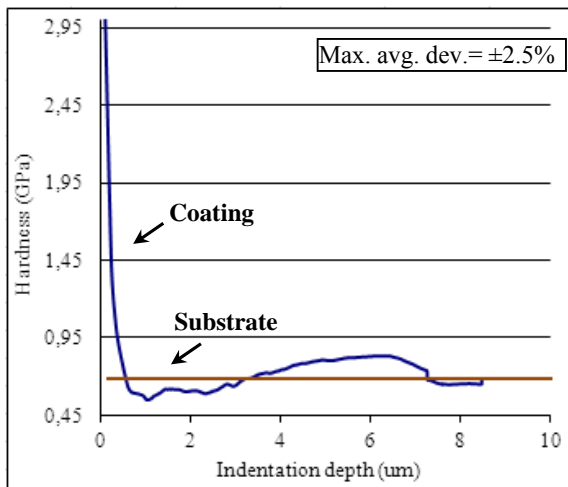


Fig. 4. Bonding strength between the substrate and coating

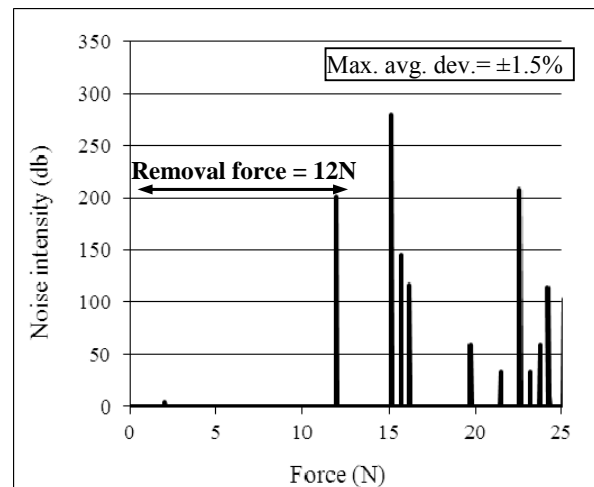


Fig. 5. Bonding strength between the substrate and coating

### c) Microstructure

Figure 6 shows the X-ray diffraction spectra of the coating on Ti substrate. It can be seen that the coating is mainly composed of rutile-anatase-brookite phase  $\text{TiO}_2$  with traces of  $\text{SiO}_2$  incorporated from electrolyte constituent  $\text{Na}_2\text{SiO}_3$ . The coating also contains some Ti elements, which must have derived from the substrate during electrochemical reaction.

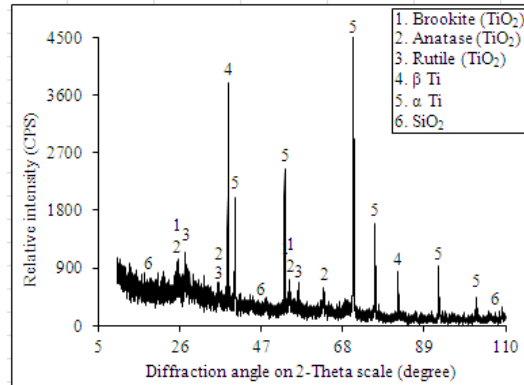


Fig. 6. Microstructure as examined in XRD

#### d) Sliding friction

Figure 7 shows the friction behavior of coating in dry and various lubrication conditions. It is to be noticed that the coating even in dry state offers 2 times less friction than the Ti substrate, which means coating tend to facilitate the forming process. The friction behavior of coating further improves when it is filled with a lubricant (see curve 2 to 5). The friction co-efficient decreases (from 0.5 to 0.2) as the proportional amount of MoS<sub>2</sub> in grease increases from 2 to 4. It is because of the fact that MoS<sub>2</sub> retains its lubricity while grease gradually melts as the sample temperature rises with time. However, as found from the tests (not shown here to avoid mess up of curves), the above finding is valid for a certain fraction of MoS<sub>2</sub>. Further increase in MoS<sub>2</sub> quantity above 4:1 (i.e., more than 80% MoS<sub>2</sub>) results in increased friction: coefficient again gradually increases from 0.2 to 0.5 as the proportion of MoS<sub>2</sub> is increased from 80% to 100% (compare curve 5 with curve 2). Therefore, at present coating lubricated with the 4:1-paste seems to be the most appropriate lubrication solution for SPIF of Ti sheet.

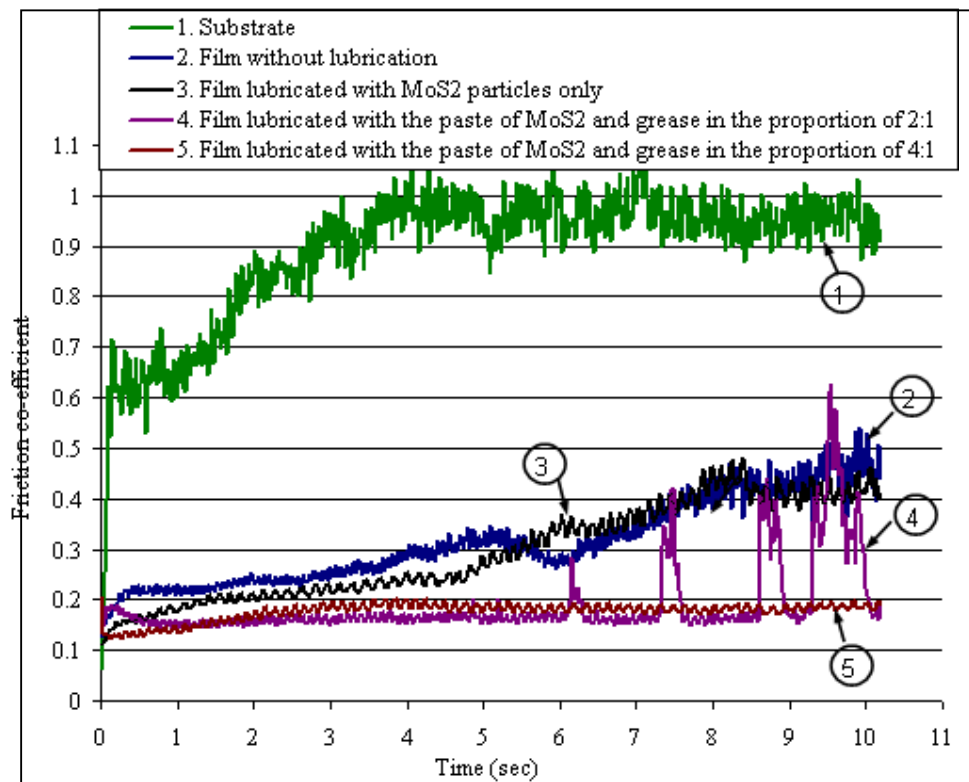


Fig. 7. Friction behavior of substrate and surface coating under different lubrication conditions

#### 4. CONCLUSION

The micro-arc oxidation process has shown promise to develop porous lubricant coating for incremental forming of pure Ti sheet [9, 10]. With an objective to standardize the coating, various characteristics of the coating were determined in this work, as summarized below:

1. The ultra-micro hardness of the coating varied from 3GPa to 0.5GPa. The coating was found to be compact between 0.8 $\mu$ m and 6.5 $\mu$ m depth where it possessed an average hardness of 0.86GPa.
2. The average bonding strength between the coating and substrate was measured to be 12N.
3. The coating was mainly composed of titanium oxide with 3 phases namely rutile, brookite and anatase.

Lubricated coating offered less friction than dry coating. The friction co-efficient of coating decreased from 0.5 to 0.2 as the proportional quantity of MoS<sub>2</sub> powder in the grease was increased. However, excessively large quantity of the powder (i.e., > 80%) resulted in increased friction. Therefore, the quantity of MoS<sub>2</sub> should be carefully controlled in order to form parts with good surface finish.

#### REFERENCES

1. Shim, M. S. & Park, J. J. (2011). The formability of aluminum sheet in incremental forming. *J Mater Process Technol*, Vol. 113, pp. 654–658.
2. Jeswiet, J., Micari, F., Hirt, G., Bramley, A., Duflou, J. & Allwood, J. (2005) Asymmetric single point incremental forming. *CIRP Ann*, Vol. 54, pp. 623–650.
3. Ham, M. & Jeswiet, J. (2006). Single point incremental forming and the forming criteria for A3003 *CIRP Ann*. Vol. 55, No. 1, pp. 241–248.
4. Hussain, G., Dar N. U., Gao, L. & Chen, M. H. (2007). A comparative study on the forming limits of an aluminum sheet-metal in negative incremental forming. *J Mater Process Technol*, Vol. 187–188, pp. 94–98.
5. Hussain, G. & Gao, L. (2007). A novel method to test the thinning limit of sheet metal in negative incremental forming. *Int J Mach Tool Manuf*, Vol. 47, pp. 419–435.
6. Jeswiet, J. & Hagan, E. (2001). Rapid prototyping of a headlight with sheet metal. *In: Proceeding of Shemet*, pp. 165–170.
7. Fratini, L., Ambrogio, G., Lorenzo, R. D., Filice, L. & Micari, F. (2002). Influence of mechanical properties of the sheet material on formability in single point incremental forming. *CIRP Ann*, Vol. 53, pp. 207–210.
8. Jackson, K. & Allwood, J. (2009). The mechanics of incremental forming. *J Mater Process Technol*, Vol. 209, pp. 1158–1174.
9. Hussain, G. & Gao, L. (2009). A fundamental investigation on the formability of a commercially-pure titanium sheet-metal in the incremental forming and stamping processes. *In: Proceedings of ASME MSEC*, pp. 943–948.
10. Hussain, G., Gao, L., Hayat, N., Cui, Z. & Pang, Y. C. (2007). Tool and lubricant for negative incremental forming of a commercially-pure titanium sheet. *J Mater Process Technol*, Vol. 203, pp. 193–201.
11. Fan, G., Gao, L., Hussain, G. & Wu, Z. (2008). Electric hot incremental forming: A novel technique. *Int J Mach Tool Manuf*, Vol. 48, pp. 1688–1692.
12. Franzen, V., Kwiatkowskia, L., Martins, P.A.F. & Tekkaya A.E. (2008). Single point incremental forming of PVC. *J Mater Process Technol*, Vol. 209, pp. 462–469.
13. Javanmard, S. A. S., Daneshmand, F., Moshksar, M. M. & Ebrahimi, R. (2011). Meshless analysis of backward extrusion by natural element method, *Iranian Journal of Science & Technology, Transaction B: Engineering*, Vol. 35, pp. 167-180.
14. Gandjalikhan, S. A. N., Shoi, H. & Zaim, E. H. (2011). Study of lubricant compressibility effect on hydrodynamic characteristics of heavily loaded journal bearings. Vol. 35, pp. 101–105.

15. Uzuna, O., Kölemena, K., Çelebi, S., Güçlü, S. & Güçlü, N. (2005). Modulus and hardness evaluation of polycrystalline superconductors by dynamic micro-indentation technique. *J Eur Cer Soc*, Vol. 25, pp. 969–977.
16. Minutolo, F. C., Durante, M., Formisano, A. & Langella, A. (2007). Evaluation of the maximum slope angle of simple geometries carried out by incremental forming process. *J Mater Process Technol*, Vol. 194, pp. 145-150.
17. Wang, Y. M., Jiang, B. L., Lei T. Q., & Guo, L. X. (2006). Microarc oxidation coatings formed on Ti6Al in Na<sub>2</sub>SiO<sub>3</sub>
18. system solution: Microstructure, mechanical and tribological properties. *Surf Coat Tech*, Vol. 201, pp. 82–89.