Nano scratch and Nanoindentation: An Approach to Understand the Tribological Behaviour of Max Phase Material Ti$_2$AlC

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Abstract. The tribological behaviour of max phase material Ti$_2$AlC was investigated by nano scratch and nanoindentation. Ti$_2$AlC samples were prepared by hot Isostatic pressing and Roughness of 0.002 µm of the Ti$_2$AlC samples were measured by Optical 3D profilometer. Nano scratch tests were conducted at low loads 2000-10000 µN to investigate the tribological property of the MAX Phase Ti$_2$AlC sample. Structural analysis and microstructure of MAX Phase Ti$_2$AlC were measured by Raman Spectroscopy and Optical microscope. The results indicate that the mechanical properties decrease with increase in load as well as dwell time and tribological properties also decrease as COF increases with increase in load.

1. Introduction

An emerging class of materials which can enable to meet the requirements of work in harsh environmental conditions and high temperature resistant are MAX Phase compounds; a group of layered ternary carbides and nitrides, where M represents early transition metals, A represents IIA or IVA group element and X represents Carbon or Nitrogen. These phases can be represented by a general formula Mn+1AXn, where n is 1, 2, or 3.

Compounds merge certain properties of metals, like electrical and thermal conductivity, resistance to thermal shock, good machinability and properties of ceramics, like high-temperature strength, oxidation, high elastic moduli, and resistance to corrosion. Many of these properties are attributed to the nano-laminated structure, with MX slabs with an interlayer of pure element [1]. The tribological properties of piston ring coated with Mo-Ni-Al alloy containing varying amount of Cr$_2$AlC MAX phase was evaluated. Relatively slow wear rate, low coefficient of friction and doubling the coating life cycles, by using MAX-phase composite coating was successfully demonstrated [2]. The increasing demands for reducing weight in automobile and aerospace industry have aroused widely attention on light weight metallic materials, such as magnesium, aluminum and titanium [3–7]. The COF of bulk Ti$_3$SiC$_2$ is 0.16 under the normal pressure of 0.8 MPa and a sliding speed of 40 m/s, which is comparable to steel disk at room temperature and dry environmental conditions (Huang et al., 2007). The average COF of bulk polycrystalline Ti$_3$AlC$_2$ is 0.1 under the normal pressure of 0.8MPa and a sliding speed of 60 m/s, which is comparable to steel disk at room temperature and dry environmental conditions (Zhai et al., 2005).

The quasi-metallic behavior of MAX phases increases the tendency for making stronger bonding with Al matrices. Despite such promising attributes, aluminum matrix composites with the contribution of MAX phases have not been systematically investigated yet. Hence, the present study is devoted to investigate the nano mechanical and nano tribological properties of sintered Ti$_2$AlC MAX phase using a Berkovich indenter with radius 50 nm by nanoindentation and nanoscratch technique at small indentation loads varying between 3000 – 9000 µN and 1000 – 4000 µN respectively.
investigation of the deformation mechanisms occurring during the scratch process has been conducted and important factors that require consideration, when using the nano-scratch test, are highlighted.

2. Experimental Procedure

2.1 Materials

Ti2AlC prefabricated samples were obtained from Dr. M W Barsoum’s laboratory, Department of mechanical science and engineering, Drexel University, USA. The samples were fabricated using Hot Isostatic Pressing technique.

2.2 Sample preparation

The obtained sample was polished with a 300, 600, 1000, 1200, 1500 and 2000-grit emery paper. Then the sample was polished by diamond paste of particle size 9, 6, 3, 1, 0.25 followed by ultrasonic cleaning in acetone for 10 min to remove the contaminants from the surface and then dried in an oven at 50 °C for 5 minutes. The roughness of the sample was measured by 3D optical profilometer.

2.3 Surface Morphology and Testing

Structural analysis and microstructure of MAX Phase Ti2AlC sample were done by Raman Spectroscopy and Optical microscope.

The depth-sensing indentation tests were performed using a nano indenter (Hysitron; TI Premier) at basic QS trapezoid loading equipped with a three-side pyramidal diamond tip (Berkovich tip with a radius of about 50nm) under ambient condition. Three peak loads 3000, 6000 and 9000 µN were used to investigate the indentation hardness and Young’s modulus as a function of penetration depth. Indentation Young’s modulus (E) and hardness (H) were calculated by the nano indenter software TriboScan version 9 based on the model of Oliver and Pharr [20]. Nanoscratch tests of 1000 – 5000 µN loads were performed on the Ti2AlC at the constant loading with a scratch length of 10 µm at room temperature to investigate the tribological property of sintered Ti2AlC sample.

3. Results and Discussion

3.1. Young’s Modulus and Hardness

Table 1 illustrates the Young’s modulus, hardness, contact depth, max. depth of Ti2AlC for a load range of 3000 - 9000 µN at dwell time of 3 sec. Young’s modulus and hardness of Ti2AlC was found between 260.21 - 60.07 GPa and 33.98 - 8.24 GPa respectively. Young’s modulus values significantly depend upon the indentation load and dwell time. The Young’s modulus is decreasing with increasing load and dwell time, owing to plastic deformation of material. The decreased Young’s modulus is also attributed to the varying slope of unloading. The hardness of Ti2AlC samples decreasing with increasing indentation load and dwell time, which shows a significant indentation load effect for hardness. Table1 shows nano mechanical properties of properties of Ti2AlC MAX Phase.

3.2. Contact Depth and Max. Depth

From the Table 1 it is clear that max. depth and contact depth are increasing with increase in load and dwell time. The dependence of the nano indentation load on depth must be justified by the relationship between load and indentation size, as indentation depth is proportional to the indentation size at applied load. The load dependence can also be described quantitatively through the application of the classical power law:

\[ P = A d^n \]  

(1)

Where \( P \) is the indentation test load and \( d \) is the resulting indentation size. The value of \( A \) and \( n \) are derived from the curve fitting of experimental results. The exponent \( n \) has been experimentally observed to be between 1 and 2. Eq. (1) is sometimes referred to as Meyer’s law. This is known as the indentation load/size effect.

3.3. Nanoscratch wear behaviour of Ti2AlC

Table 2 shows the nano-scratch properties of Ti2AlC MAX Phase. Figure 1 shows the 2D and 3D images of scratch on Ti2AlC sample at 1000 µN, 2000 µN, 3000 µN and 4000 µN. Figure 2 shows the
graph which indicate that the scratch depth increases with the increasing load and the scratch depth as a function of normal load. The increase in scratch depth with increase in load is due to increase in the contact area between the indenter tip and the Ti2AlC sample surface. Moreover, the scratch depth matches well with the hardness results, as hardness decreases with increase in load. It is clear from Figure 1 that the scratch images shows smooth scratch path with no cracks and debris on the surface. Moreover, material flow was plastic which is displaying by a layered piling up of material at higher loads. Thus, the wear mechanism at room temperature is mainly brittle and abrasive which results in increasing COF as shown in Figure 3. The dependence of COF with time of Ti2AlC Max phase is shown in Figure 4. It is clear from Figure 4 that with the increase in time COF remains almost constant due to the lubricating property of the MAX phase ceramics.

4. Conclusion
In this study, we studied the nanomechanical and nanotribological properties of the Ti2AlC MAX phase. Several major conclusions were obtained as follows:
1. The mechanical properties of Ti2AlC decreased with increase in load as well as dwell time.
2. The tribological properties of Ti2AlC decreased as COF increases with increase in load.
3. The scratch depth increased with the increasing load.
4. The wear mechanism was abrasive and brittle which resulted in increasing COF with increasing load.

Table 1. Nano-mechanical properties of properties of Ti2AlC MAX Phase.

| S.No. | Load (µN) | Reduced Modulus (GPa) | Hardness (GPa) | Contact Depth (nm) | Max. Depth (nm) |
|-------|-----------|-----------------------|----------------|--------------------|-----------------|
| 1.    | 3000      | 260.2                 | 30.88          | 53.1               | 77.7            |
| 2.    | 6000      | 193.30                | 26.19          | 86.3               | 129.4           |
| 3.    | 9000      | 129.10                | 11.87          | 166.4              | 219.6           |

Table 2. Nano-scratch properties of Ti2AlC MAX Phase.

| S. No. | Load (µN) | Type of Loading | Scratch Length (nm) | COF | Scratch Depth (nm) |
|--------|-----------|-----------------|---------------------|-----|--------------------|
| 1.     | 1000      | Constant Load Scratch | 10              | 0.11 | 27.06              |
| 2.     | 2000      | Constant Load Scratch | 10              | 0.15 | 39.19              |
| 3.     | 3000      | Constant Load Scratch | 10              | 0.22 | 46.20              |
| 4.     | 4000      | Constant Load Scratch | 10              | 0.22 | 63.59              |
Figure 1. The 2D and 3D images of Scratch on Ti2AlC sample at 1000 µN (a,b) 2000 µN (c,d) 3000 µN (e,f) 4000 µN (g,h)

Figure 2. The dependence of scratch depth of Ti2AlC on load.
Figure 3. Effect of load on COF during scratching

Figure 4. The dependence of COF of Ti$_2$AlC with time.

References

[1] W.B. Tian, P.L. Wang, Y.M. Kan, G.J. Zhang 2008 J. Mater. Sci. 43 2785–2791.

[2] D. Davis, M. Srivastava, M. Malathi, B. B. Panigrahi, S. Singh 2018 Applied Surface Science 49 295–303.

[3] W.J. Joost, P.E. Krajewski 2017 Scr. Mater. 128 107–112.

[4] P. Singh, H. Pungotra, N.S. Kalsi 2017 Mater. Today Proceed. 4 8971–8982.

[5] L. Tian, A. Russell, T. Riedemann, S. Mueller, I. Anderson 2017 Mater. Sci. Eng. A 690 348–354.
[6] H. Friedrich, S. Schumann 2001 J. Mater, Process. Technol. 117 276–281.

[7] W. Yu, Y. Cao, X. Li, Z. Guo, S. Xiong 2017 J. Mater. Sci. Technol. 33 52–58.

[8] M.W. Barsoum 2000 Prog. Solid State Chem. 28 201-281.

[9] Z.M. Sun 2011 Int. Mater. Rev. 56 143-166.

[10] M. Radovic, M.W. Barsoum 2013 Am. Ceram. Soc. Bull. 92 20-27.

[11] T. A. Prikhnaa, S. N. Dub, A. V. Starostina, M. V. Karpets, T. Cabiosh, and P. Chartier, 2012 Journal of Superhard Materials 34 2

[12] Z. Sun 2011 Int. Mater. Rev. 56 143–166.

[13] M.W. Barsoum, D. Brodkin, T. El-Raghy 1997 Scr. Mater. 36 535–541.

[14] M. Barsoum, T. El-Raghy, M. Ali 2000 Metall. Mater. Trans. A 31 1857–1865.

[15] M. Krinitcyn, Z.W. Fu, J. Harris, K. Kostikov, G.A. Pribytkov, P. Greil, N. 2017 Ceram. Int. 43 9241–9245.