Nanoindentation characterization of surface layers of electrical discharge machined WC–Co

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Abstract

This study applies nanoindentation and other analysis techniques to investigate the influence of wire electrical discharge machining (EDM) process on the structure and properties of machined surface layers of WC–Co composites. Multiple indents were conducted on the cross-section of the surface recast layer, sub-surface heat-affected zone, and bulk material. The energy disperse X-ray spectrometry and X-ray diffraction were used to analyze the material compositions in the heat-affected zone and recast layer and to study the electrical spark eroded surface. The indents were inspected by scanning electron microscopy to distinguish between regular and irregular indents in these three regions. Irregular indents were caused by the porosity, soft matrix material, separation of grain boundaries, and thermal cracks caused by EDM process. The hardness and modulus of elasticity obtained from regular indents in bulk material and heat-affected zone were comparable to those of WC. It was found that the recast layer had lower hardness and modulus of elasticity than the bulk material and heat-affected zone.

Keywords: Nanoindentation; Mechanical properties; Metal matrix ceramic composites; Electrical discharge machining

1. Introduction

Electrical discharge machining (EDM) is a thermo-electric process that erodes workpiece material by a series of discrete electrical sparks between the workpiece and electrode flushed by or immersed in a dielectric fluid. Unlike traditional cutting and grinding processes which rely on a much harder tool or abrasive material to remove the softer work-material, the EDM process utilizes electrical sparks or thermal energy to erode the unwanted work-material and generate the desired shape. The hardness and strength of the work-materials are no longer the dominating factors that affect the tool wear and hinder the machining process. This makes EDM particularly suitable for machining hard, difficult-to-machine materials, such as metal matrix composites (MMCs). As shown in Fig. 1(a), the conventional wire EDM process uses a traveling wire, ranging from 0.01 to 0.36 mm in diameter, as the electrode to erode a groove in the workpiece. The close-up view of the gap and electrical sparks between the wire and workpiece is illustrated in Fig. 1(b). The cylindrical wire EDM process, as shown in Fig. 1(c), adds a rotary axis to enable the generation of cylindrical forms [1,2]. The mechanical properties of surface layers generated in this process are critical to the part performance, particularly its fatigue and wear properties.

MMC materials are widely used because of their outstanding hardness, wear resistance, and strength. Although, MMCs are difficult to machine by conventional processes due to the high hardness and toughness, the EDM process has been used extensively and successfully to manufacture MMC components [1–4]. EDM can machine precise, complex, and intricate MMC components. A surface recast layer and a subsurface heat-affected zone are generated [5]. On the
EDM surfce, the surface integrity, including roughness, size of craters, and depth of a recast layer and heat-affected zone, on cylindrical wire EDM WC\textsubscript{C}/C\textsubscript{1}/Co surface has been investigated \([2]\). This study applies nanoindentation, energy dispersive spectroscopy (EDS), and X-ray diffraction methods to farther study the mechanical properties (hardness and modulus of elasticity) and material compositions of the WC\textsubscript{C}/C\textsubscript{1}/Co surface layers machined by the cylindrical wire EDM process.

Fig. 2 shows scanning electron microscopy (SEM) micrographs of the surfaces and cross-sections of WC--Co samples machined under 5 and 14 ms pulse on-time, which is defined as the duration of the high voltage of an electrical spark cycle \([2]\). Electrical sparks generate craters, bubbles, and cracks on the recast layer, marked by RL. A sub-surface heat-affected zone, denoted by HA, with essentially no porosity can be identified between the recast layer and bulk material. Pulse on-time is an important EDM process parameter that affects the thickness of the recast layer and heat-affected zone \([2]\). Keeping the spark cycle at 28 ms, the longer (14 ms) pulse on-time generates thicker recast layer and heat-affected none, as shown in Fig. 2(a). The material removal rate is, in general, higher for the longer pulse on-time. The sample machined by the shorter (5 ms) pulse on-time, as shown in Fig. 2(b), was selected for the nanoindentation and other material characterization studies.

Indentation has been widely used as an experimental tool to probe the hardness and modulus of materials by means of load-displacement behavior. Nanoindentation applies very small loads, at the \(\mu\text{N}\) level, to the specimen and generates indentation depths in the sub-\(\mu\)m or nm scale \([6]\). This makes nanoindentation suitable and effective for measuring mechanical properties of thin films and small volumes of material \([7--12]\). The thickness of EDM recast layer and heat-affected zone is usually very thin \([2--4]\). For example, as shown in Fig. 2, the thickness of EDM surface layers is less than 3 \(\mu\)m. It is difficult to use conventional methods to measure and distinguish the mechanical properties of EDM surface layers. Llanes et al. \([3]\) used microscratch tests to characterize the sliding contact response of the wire EDM WC--Co surfaces. In this study, the nanoindentation method is applied.

The WC--Co material is not homogeneous. The cross-section surface consists of the hard, 1--2 \(\mu\)m, WC grain in the size close to the nano-indents and soft Co matrix. Porosity, about 0.5 \(\mu\)m in size, can also be seen on the polished cross-section surface in Fig. 2. The nanoindentation results are highly influenced by the composition and defect of the material near the indent. One of the goals of this study is to correlate the indentation results with the SEM micrographs of individual nano-indents. Regular nano-indents are identified and compared.
The experiment setup and sample preparation are introduced in the following section. Results of nanoindentation and EDS X-ray and X-ray diffraction analysis of EDM surface layers are presented in Section 3.

2. Experiment setup and sample preparation

In the past two decades, the nanoindentation method with continuous load-displacement sensing has been developed and applied by researchers to measure mechanical properties of thin films and surface layers. The hardness, $H$, and modulus of elasticity, $E$, can be calculated using the following formulas.

$$H = \frac{P_{\text{max}}}{A}$$  \hspace{1cm} (1)

$$E_i = \sqrt{\frac{S}{2}} \frac{\sqrt{A}}{\sqrt{A}}$$  \hspace{1cm} (2)

$$1 = \frac{(1 - v^2)}{E} + \frac{(1 - v^2)}{E_i}$$  \hspace{1cm} (3)

where $P$ is the indentation load and $P_{\text{max}}$ is the peak indentation load, $h$ is the displacement of the indenter, $A$ is the projected area of the hardness impression, $S (= dP/dh)$ is the contact stiffness, $\nu$ is the Poisson’s ratio of the work-material, $E$ is the modulus of elasticity of the work-material, and $E_i$ is the reduced modulus of elasticity with consideration of the effect of non-rigid indenters. $E_i$ and $v_i$ are the modulus of elasticity and Poisson’s ratio of the indenter, respectively.

In this study, the continuous stiffness measurement method was used to obtain the contact stiffness, $S$. Details of this technique and analysis procedure are described in Refs. [6,13,14]. The displacement control was used in the nanoindentation experiment and the load was dependent on the indenter penetration depth. The hardness and modulus of elasticity were calculated from the load-displacement curves.

The nanoindentation experiments were conducted at the Oak Ridge National Laboratory on a MTS Nanoindenter™ II with a Berkovich diamond indenter. The machine, as shown in Fig. 3, was operated to measure the surface properties of WC–Co after EDM. A precision $X$–$Y$ table with a calibrated distance from the indenter to an optical microscope was used for accurate positioning of the indents.

The WC–Co composite used in this study consists of WC particles with a volume fraction of 10% cobalt matrix. It has 92 Re hardness, 3.4 GPa transverse rupture stress, and 14.5 specific density. The WC–Co cylindrical part was machined using a Brother HS-5100 wire EDM machine with 0.25 mm diameter brass wire, 55 V gap voltage, 28 $\mu$s spark cycle time, 5 $\mu$s pulse on-time, 15 mm s$^{-1}$ wire axial speed, and 17.6 N wire tension. The wire electrode, traversing at 0.5 mm min$^{-1}$, was used to cut a WC–Co rod spinning at 50 rpm from 6.35 to 5.08 mm in diameter. Detailed EDM process parameters were given in Ref. [2]. The cross-section of the EDM surface layers of this WC–Co rod, as shown in Fig. 2(b), was used for the nanoindentation tests.

3. Experimental results and discussion

Multiple indents were performed on the cross-section of the bulk material, heat-affected zone, and recast layer of the WC–Co part machined by cylindrical wire EDM. Examples of three sets of nano-indents on the cross-section surface are shown in Fig. 4. The ten nano-indents in the bulk material are identified B1–B10 in Fig. 4(a). Four nano-indents in the heat-affected zone are labeled by H1–H4 in Fig. 4(b). R1–R6 are the six nano-indents in the recast layer.

The calculated hardness and modulus of elasticity of the 14 sample nano-indents from each of the three regions (bulk material, heat-affected zone, and recast layer) are presented in Table 1. These data are arranged in descending order of hardness values. Relatively large variations of both the calculated hardness and modulus of elasticity were obtained in all three regions. The porosity, soft cobalt matrix, cracks, and defects within three regions contributed to the large discrepancy in the experimentally determined hardness values. This will be further illustrated by SEM micrographs of sample nano-indents in each of three regions in the following three sections.

3.1. Bulk material

The porosity and soft Co matrix surround the hard WC grain can be identified on the cross-sectional SEM micrographs of the EDM surface of WC–Co. For the 14 nano-indents in the bulk material listed in Table 1, the calculated hardness varied between 7.6 and 19.7 GPa and the modulus of elasticity ranged from 239 to 514 GPa. These indents were examined using SEM and then classified into two groups: regular and irregular nano-indents. The irregular nano-indents have pores and/or separation of grain boundaries on the indent area resulting in lower values of hardness and modulus of elasticity.

SEM micrographs of two sample nano-indents from each group are shown in Fig. 5. One sample load-displacement curve for one of the nano-indents in each set of frames is also shown in Fig. 5.

3.1.1. Regular nano-indents

Fig. 5(a) shows two regular nano-indents located on the WC particles. The calculated hardness values are...
19.7 and 19.3 GPa, respectively. This level of hardness is comparable to the value of 18 GPa that has been reported for WC [12]. In contrast to irregular indents, which hit pores or generated separation of grain boundaries, these two indented areas had only minor damage on the outside edges.

3.1.2. Irregular nano-indents
Two typical irregular nano-indents are shown in Fig. 5(b). A large portion of the indent with 7.7 GPa hardness was obtained on a region with pores. The indent with 11.6 GPa hardness had significant separation grain boundaries around the center, possibly due to a pore underneath the surface. For these two irregular indents, the contact area was difficult to define because of the morphology, which resulted in lower values of hardness and elastic modulus.

3.2. Heat-affected zone
The heat-affected zone is located between the recast layer and the bulk material. For the WC–Co composite, the Co melted and resolidified in the heat-affected zone during the EDM spark erosion and filled the pores between the WC particles. As shown in Fig. 2 and Fig. 6, this can be observed in SEM micrographs of the WC–Co cross-section and has been used to identify the depth of heat-affected zone. The thickness of the heat-affected zone in this sample is about 1–2 μm.

Unlike nano-indents in the bulk material, separation of grain boundaries rarely occurred during nanoindentation in the heat-affected zone. Very likely the cobalt filled the pores surrounding the WC particles. Variations of calculated hardness and modulus of elasticity in the heat-affected zone are lower than those in the bulk material and recast layer. The calculated hardness varied between 15.0 and 20.4 GPa, and the modulus of elasticity ranged from 323 to 515 GPa.

The nano-indents in the heat-affected zone can also be grouped into regular nano-indents and irregular nano-indents.

3.2.1. Regular nano-indents
Fig. 6(a) shows two regular nano-indents with hardness of 20.4 and 18.1 GPa. Similar to the regular nano-indents in bulk material, no pore or separation of grain
boundaries is close to the center of the nano-indent. The calculated results reflect the mechanical properties of WC.

3.2.2. Irregular nano-indents

Fig. 6(b) shows two samples of irregular nano-indents and a large overview of a thermal crack extending from the surface, through the recast layer, to an indent in the heat-affected zone. This study showed that irregular nano-indents are less likely to occur, and smaller variation of properties are observed in the heat-affected zone. This is most likely due to the pores being filled by the melted and resolidified Co matrix. The softness of the Co matrix is one possible reason for the irregular indents in the heat-affected zone. Another possibility is the indent on a thermal crack initiated from outside the heat-affected zone, as shown in the 15.0 GPa hardness indent in Fig. 6(b).

3.3. Recast layer

The recast layer is defined as the material melted by electrical sparks and resolidified onto the surface without being ejected nor removed by flushing. As shown in Fig. 2 and Fig. 7, the grain boundaries of WC are not

Table 1
Nanoinindentation hardness and modulus of elasticity of bulk material, heat-affected zone, and recast layer (in descending order of the hardness) (Unit: GPa)

<table>
<thead>
<tr>
<th>Bulk material</th>
<th>Heat-affected zone</th>
<th>Recast layer</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Hardness</strong></td>
<td><strong>Modulus</strong></td>
<td><strong>Hardness</strong></td>
</tr>
<tr>
<td>19.7</td>
<td>497</td>
<td>21.0</td>
</tr>
<tr>
<td>19.3</td>
<td>496</td>
<td>20.4</td>
</tr>
<tr>
<td>17.4</td>
<td>514</td>
<td>19.9</td>
</tr>
<tr>
<td>17.4</td>
<td>479</td>
<td>19.3</td>
</tr>
<tr>
<td>16.5</td>
<td>453</td>
<td>18.5</td>
</tr>
<tr>
<td>16.2</td>
<td>493</td>
<td>18.2</td>
</tr>
<tr>
<td>13.9</td>
<td>417</td>
<td>18.1</td>
</tr>
<tr>
<td>13.8</td>
<td>462</td>
<td>17.9</td>
</tr>
<tr>
<td>13.2</td>
<td>477</td>
<td>16.5</td>
</tr>
<tr>
<td>12.3</td>
<td>477</td>
<td>16.4</td>
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<tr>
<td>11.6</td>
<td>401</td>
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<tr>
<td>7.7</td>
<td>238</td>
<td>15.0</td>
</tr>
<tr>
<td>7.6</td>
<td>395</td>
<td>15.0</td>
</tr>
</tbody>
</table>

Regular nano-indents are indicated in ‘bold type’ and others are irregular nano-indents.

Fig. 5. Nanoinindentation in the bulk material. (a) Regular nano-indents. (b) Irregular nano-indents. Legend: S, Separation of grain boundaries; P, Pore.

Fig. 6. Nanoinindentation in heat-affected zone. (a) Regular nano-indents. (b) Irregular nano-indents. (c) Large view showing the thermal crack extended from the recast layer.
clear and the porosity disappeared in the recast layer. Micro-bubbles and cracks, generated by thermal stresses in cylindrical wire EDM process, exist in the recast layer. The thickness of the recast layer of the sample examined in this experiment is about $2\times10^{-3}$ mm.

The calculated hardness and modulus of elasticity of the recast layer are generally lower than those of the bulk material and heat-affected zone. The deposition of brass from the wire electrode and oxidation products, identified by EDS X-ray and X-ray diffraction later in this section, and thermal cracks are the main causes for the deterioration of mechanical properties in the recast layer. Regular and irregular nano-indents in the recast layer were also identified.

(1) Regular nano-indents: Fig. 7(a) shows two regular nano-indents in the area without clear grain boundary of WC, pores, and cracks. The calculated hardness and modulus of elasticity were lower than the values obtained from regular indents in the bulk material and heat-affected zone.

(2) Irregular nano-indents: The calculated hardness and modulus of elasticity were highly affected by thermal cracks, bubbles, and edges in the recast layer. Some indents with very low hardness and modulus of elasticity were obtained, as shown in Fig. 7(b).

EDS X-ray was used to identify the elements in three regions: bulk material, heat-affected zone, and recast layer. The bulk material and heat-affected zone had similar EDS X-ray analysis results, as shown in Fig. 8(a), that W, Co, and C were present in the two regions. Fig. 8(b) shows that three additional elements, Cu, Zn, and O, were detected in the recast layer. This can be explained by the melting and resolidification of the brass wire electrode during EDM spark erosion. Oxidation also occurs a result of the high temperature involved in this process, hence the presence of oxygen. Such contamination of Cu, O, and Zn on the surface of wire EDM silicon has been reported by Uno et al. [15] using X-ray photoelectron spectroscopy.

X-ray diffraction was then used to further analyze the material composition of the recast layer. A 4-axis ($\Phi, \chi, \Omega, 2\Theta$) goniometer [16] using copper radiation was employed for this analysis. Table 2 lists the details of the experimental conditions for the X-ray diffraction measurements. The X-ray diffraction results are shown in Fig. 9. Major peaks in the scan revealed that the recast layer contains tungsten carbide (WC, $W_2C-x$), and $W_2C$, brass (Cu, Zn), and cobalt oxide (CoO). Copper oxide (CuO) and/or tungsten oxide carbide ($W_2(C, O)$), possibly exist in the recast layer, but could not be distinguished. These results indicate that tungsten carbide is still present in the recast layer, even though the corresponding grain boundaries are indistinct. The presence of Cu and Zn results from the melting of the brass wire electrode and subsequent deposition in the surface recast layer. Oxides were likely generated due to the high temperature in EDM spark erosion.

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Experimental conditions of the X-ray diffraction measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Parameter</td>
<td>Condition</td>
</tr>
<tr>
<td>Equipment</td>
<td>Scintag PTS goniometer MAC Science</td>
</tr>
<tr>
<td>Power</td>
<td>18 kW rotating anode generator Scintag</td>
</tr>
<tr>
<td>Radiation</td>
<td>Cu, $\lambda = 1.54059$ Å, line mode</td>
</tr>
<tr>
<td>Incident optic</td>
<td>Cu, $\lambda = 1.54059$ Å, line mode</td>
</tr>
<tr>
<td>Incident slit width</td>
<td>0.5 mm</td>
</tr>
<tr>
<td>Receiving slit</td>
<td>0.25°; radial divergence limiting (RDL) Soller</td>
</tr>
<tr>
<td>Source to specimen distance</td>
<td>360 mm</td>
</tr>
<tr>
<td>Specimen to back slit distance</td>
<td>280 mm</td>
</tr>
<tr>
<td>Scans</td>
<td>$\Theta-2\Theta$; 0.02° $2\Theta$ step; 0.2°/min</td>
</tr>
</tbody>
</table>
4. Concluding remarks

Mechanical properties of the bulk material, subsurface heat-affected zone, and surface recast layer of a WC-Co part machined by cylindrical wire EDM were investigated by means of nanoindentation. SEM was used to distinguish between regular and irregular indents. Irregular indents were caused by porosity, softness of the Co matrix, thermal cracks, and separation of grain boundaries generated during indentation. The values of hardness and modulus of elasticity obtained from the regular indents in the bulk material and heat-affected zone are comparable to those of WC.

More compact microstructure and better support of the WC particles was observed in the heat-affected zone. For regular indents, the recast layer had lower hardness and modulus of elasticity than the bulk material and heat-affected zone. The grain boundaries of WC could not be observed in the recast layer, though EDS X-ray and X-ray diffraction showed that WC still existed in the recast layer. In addition, Cu and Zn from the brass wire electrode, and oxides of Cc and Cu as a result of high temperature involved in the EDM process were identified in the recast layer. These effects and thermal cracks and bubbles all contribute to the deterioration of mechanical properties of the recast layer.

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