Comparison of Nano-Mechanical Behavior between Selective Laser Melted SKD61 and H13 Tool Steels

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Abstract: Using nanoindentation under various strain rates, the mechanical properties of a selective laser melted (SLM) SKD61 at the 800 mm/s scan speed was investigated and compared to SLM H13. No obvious pile-up due to the ratio of the residual depth (h_f) and the maximum depth (h_max) being lower than 0.7 and no cracking were observed on any of the indenter surfaces. The nanoindentation strain-rate sensitivity (m) of SLM SKD61 was found to be 0.034, with hardness increasing from 8.65 GPa to 9.93 GPa as the strain rate increased between 0.002 s\(^{-1}\) and 0.1 s\(^{-1}\). At the same scan speed, the m value of SLM H13 (m = 0.028) was lower than that of SLM SKD61, indicating that the mechanical behavior of SLM SKD61 was more critically affected by the strain rate compared to SLM H13. SLM processing for SKD61 therefore shows higher potential for advanced tool design than for H13.

Keywords: Selective laser melting, SKD61 tool steel, nanoindentation, strain-rate sensitivity.

INTRODUCTION

SKD61 and H13 are types of hot work tool steels applied to casting molds, extrusion tools, forging dies, etc., and the hardness of SKD61 (229 BHN) is lower than that of H13 (235 BHN) [1-4]. Both steels are fabricated by conventional methods requiring expensive dedicated tools, and thus are inappropriate for small-scale or complex-shape productions [1-3]. Selective laser melting (SLM), is a laser powder-bed additive manufacturing process, that is suitable for processing of tool steels including SKD61 and H13 because it offers the ability to not only reduce the amount of machining and hence wastage of this expensive material, but also to produce intricate molds with a nearly full density and a refined microstructure [2,3].

The mechanical behavior of hot work tool steels prepared by the SLM method is one of the most important characteristics. It was reported that the hardness of SLM SKD61 fabricated from commercial powders was higher than that made from gas atomized spherical powders [3]. Very recently, a few studies have addressed the mechanical properties of tool steel processed by SLM using nanoindentation [2]. For example, the H13 SLM-ed at 200 mm/s scan speed exhibited the lowest creep resistance and highest hardness values. However, very few studies have dealt with the mechanical behavior of SLM SKD61 using nanoindentation. It is thus necessary to perform
nanoindentation tests to probe the creep behavior of the SLM SKD61 and compare it with that of SLM H13.

In a previous study, SLM processing at 800 mm/s laser scan speed was applied successful for SKD61 powders [3]. This study expands on that reported findings by using nanoindentation tests to investigate the mechanical properties of SLM SKD61. Results for SLM-processed H13 at the same laser scan speed sourced from the literature are used in the evaluation conducted here [2]. It is demonstrated that nanoindentation strain-rate sensitivity of the SLM SKD61 is more critical to strain rate compared to SLM H13.

EXPERIMENTAL PROCEDURE

The material used in this work was SKD61 powder prepared by a gas atomizing process (Hot Gas Atomization System, HEMMIGA 100/25, PSI Ltd., UK), as shown in Fig. 1. Specifically, SKD61 ingot obtained from SeAH Special Steel Corp. (Korea) was melted into a graphite crucible at 1690 °C under a high purity argon atmosphere. The alloy liquid was then ejected through a spray nozzle under hot N2 gas pressure of 50 bar. Upon gas atomization, the gas-atomized powders were collected and loaded onto a series of ASTM E11 standard sieves to obtain a specific particle size range of 10–45 μm. The elemental chemical compositions of the produced powders were confirmed by inductively coupled plasma (ICP, Spectro Arcos ICP-AES, Germany). The powders were then processed by a SLM device (a Concept LaserMlab-Cusing system, Germany) equipped with a 90 W Nb:YAG fiber laser to manufacture cuboid specimens (10 × 10 × 10 mm). Detailed preparation of the SLM samples was described in [2,3].

The cuboid specimens prepared by SLM were mounted in epoxy resin, cross-sectioned and then mechanically polished on an auto-disc polishing machine (Struers LaboForce-100, Denmark) to
reach mirror-like surfaces. The specimens with a surface roughness of lower than 50 nm determined using a specialized measuring instrument (Mitutoyo, SV-3200S4, Japan) were used for the next steps. An optical microscope (OM, Nikon ECLIPSE MA200, Japan) and a scanning electron microscope (SEM, JSM-5800, JEOL, Japan) were also used to observe the microstructure on the sample surfaces. Nanoindentation tests were then performed for those samples at room temperature at the same maximum load (500 mN) on a NanoTest nanoindenter (Micro Materials Ltd., Wrexham, UK) using a three-sided Berkovich diamond indenter. Specifically, all nanoindentation tests were carried out at loading rates of 50 mN/s, 25 mN/s, 16.67 mN/s, 12.5 mN/s, 10 mN/s, 5 mN/s and 1 mN/s. The indenter was then held at the maximum load for 5 s, which was followed by unloading at a rate of 50 mN/s for all tests. At least ten indentation points at each loading rate were carried out and the results were averaged.

RESULTS AND DISCUSSION

Figure 2a shows a microstructure result of the SKD61 sample prepared by SLM at 800 mm/s scanning speed, in where the inset is the SEM result. Fig. 1b shows a SEM result at the location of an indenter point. Fig. 2a reveals some black pores with irregular shapes and small volumes observed over the sample. The volume fraction of the black phase was calculated using the image analysis software to be 1.4%, which is lower than that (5.2%) for SLM H13 at the same scan speed [2]. Black pores, indicating imperfections in the microstructure, may stem from insufficient laser energy to melt powder materials within the melt pool, causing a short cooling (faster solidification) time and insufficient melting of the substrate and powders [2].

![Fig. 2. (a) Microstructure of the SLM-processed SKD61 at 800 mm/s scanning speed; (b) SEM graph of an indenter point after nanoindentation tests](image)

The widely used Oliver and Pharr method is accurate only for nanoindents that do not show significant pile-up deformation, which is related to the ratio of the residual depth ($h_r$) and the maximum depth ($h_{\text{max}}$) [5]. In the SEM results of indenter points on all sample surfaces, no obvious pile-up is observed, as shown in Fig. 1b. This result is similar to that of SLM H13 material, where it was confirmed that $\frac{h_r}{h_{\text{max}}}$ in all nanoindentation tests was lower than 0.7 [2]. Therefore, the Oliver and Pharr method can be adopted to extract the hardness values of the material here. Moreover, no cracking in Fig. 1b is observed, indicating that the material has low crack sensitivity.

In this study, a constant rate of loading (CRL) method, where a steady loading rate is used until the tip depth rate becomes nearly constant, is used to determine the strain rate ($\dot{\varepsilon}$) because the CRL allows for simple calculation of strain rates, and also is proven to be more suitable to correlate with the conventional constant strain rate tests [6]. In fact, both indentation hardness and strain rate are
not constant under CRL loading, causing difficulty in determining of a consistent creep exponent from a single CRL nanoindentation experiment [6]. However, if only the indentation hardness and indentation strain rate at the maximum load point are used, the strain-rate sensitivity can be determined from a group of CRL nanoindentation tests under different loading rates [6]. Accordingly, the above loading rates correspond to strain rates of 0.1 s\(^{-1}\), 0.05 s\(^{-1}\), 0.033 s\(^{-1}\), 0.025 s\(^{-1}\), 0.02 s\(^{-1}\), 0.01 s\(^{-1}\), and 0.002 s\(^{-1}\), respectively.

Figure 3 is plotted to compare the hardness of SKD61 and H13 processed by SLM at 800 mm/s scan speed. Error bars on the data reflect the standard deviation calculated for hardness from multiple indentations for each sample. Experimental results from nanoindentation tests of H13 prepared by SLM at 800 mm/s, obtained from another report in the literature, are included for comparison [2]. As shown in Table 1, the hardness increases in ranges of (8.65–9.99) GPa and (7.91–8.84) GPa for the SKD61 and H13 materials, respectively, prepared by SLM at the same scan speed of 800 mm/s at nanoindentation strain rates in the range of 0.002 s\(^{-1}\) and 0.1 s\(^{-1}\). This shows the SLM SKD61 has about 10% higher hardness values on average than the SLM H13 material.

Table 1. Hardness values of SKD61 and H13 prepared by the SLM process at the same scan speed of 800 mm/s

<table>
<thead>
<tr>
<th>Strain rate (s(^{-1}))</th>
<th>Hardness (GPa)</th>
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<tbody>
<tr>
<td></td>
<td>SKD61</td>
</tr>
<tr>
<td>0.002</td>
<td>8.65</td>
</tr>
<tr>
<td>0.01</td>
<td>9.26</td>
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<tr>
<td>0.02</td>
<td>9.43</td>
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<tr>
<td>0.025</td>
<td>9.26</td>
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<tr>
<td>0.033</td>
<td>9.55</td>
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<tr>
<td>0.05</td>
<td>9.70</td>
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<tr>
<td>0.1</td>
<td>9.93</td>
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By assuming the indentation strain rate and hardness are proportional to the flow strain rate and stress, respectively, it follows that the strain rate sensitivity (SRS) can be defined as

\[ m = \left( \frac{\partial \ln H}{\partial \ln \varepsilon} \right) \]  

The slope of the straight line in Fig. 3 represents the strain-rate sensitivity exponent (\(m\)), with a value of 0.034 and 0.028 for the SLM SKD61 and H13, respectively. The strain-rate sensitivity of SLM SKD61 is higher than that of SLM H13, indicating that the mechanical behavior of H13 material is less susceptible to the strain rate than SKD61.
Fig. 3. Indentation stress as a function of strain rate for SLM-processed by SKD61 and H13

In recent studies, the optimal laser scan speed for the SLM process is around 200 mm/s for H13 [2], and the hardness increased from 8.61 GPa to 9.29 GPa, and these values are lower than those of SKD61 in the present study at the same strain rate range. Because the laser energy density is inversely proportional to the scan speed [7], the optimal condition of the SLM process for SKD61 consumes less energy than for H13. Moreover, SLM SKD61 shows better microstructure (less pores) and higher hardness than SLM H13. Therefore, SLM SKD61 shows higher potential for advanced tool design than SLM H13.

CONCLUSION

This study demonstrates successful printing of SKD61 by the SLM method at 800 mm/s laser speed. From the results, no obvious pile-up (due to $\frac{h_f}{h_{max}} < 0.7$) and no cracking on all indenter surfaces were observed. Hardness increased from 8.65 GPa to 9.93 GPa as the strain rate increased in a range of 0.002 s$^{-1}$ and 0.1 s$^{-1}$. At the same scan laser speed, the $m$ value of SLM SKD61 ($m = 0.034$) is higher than that of SLM H13 ($m = 0.028$), indicating that the mechanical behavior of the SLM SKD61 was more critical to the strain rate compared to SLM H13. As a result, SLM processing for SKD61 shows higher potential in practical application than for H13 due to the superior microstructure and mechanical behavior of the former, and less laser energy consumption at the optimal condition.

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REFERENCES


