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Development of Surface Treatment Solutions for Stamping Tools Fabricated via Additive Manufacturing

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Materials Science and Technology Division
Manufacturing Science Division

DEVELOPMENT OF SURFACE TREATMENT SOLUTIONS FOR STAMPING TOOLS FABRICATED VIA ADDITIVE MANUFACTURING

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ABSTRACT

Oak Ridge National Laboratory (ORNL) and H.E.F. USA, Inc. (HEF) partnered to develop wear resistant surface treatment of additive manufactured steels for stamping die applications under CRADA agreement NFE-19-07909. This project aimed at evaluating the ARCOR® process, developed by HEF, to improve the surface behavior of 410SS (stainless steel), 410NiMo SS, 630 SS, and Maraging 250 steel coupons and demo components, fabricated via wire arc additive manufacturing (WAAM), with and without post print heat treatment. The peak hardness achieved was ~1000 HV and above (Up to 1300 HV) for all materials under proper processing parameters, and far exceeded the target hardness of 746 HV (60 HRC). Finally, stamping tools were printed and nitrocarburized with the optimum parameters, and then went through 500 fabrication cycles. Characterization of the used 17-4PH stamping tool discovered no crack or delamination in the nitrocarburized layer and the interface between the nitrocarburized layer and substrate.

1. BACKGROUND

HEF Group offers innovative solutions for wear, friction, and corrosion reduction through a diverse selection of surface treatments and hard coatings. HEF partners with industry's largest and most demanding manufacturers to develop application-specific surface engineering processes & products that substantially enhance performance and long-term durability. HEF utilizes a diverse range of surface modification technologies to deliver solutions that maximize the sought-after performance characteristics of the mechanical components under consideration. By focusing HEF's substantial global resources – people, technology, & infrastructure – on specific applications, HEF creates optimized solutions with the greatest possible value to the end-user.

HEF group currently offers surface treatment and thin-film coating solutions to minimize wear, corrosion and friction of engineered industrial machinery and automotive components. However, in the future offering tribology solutions for tooling utilized for metal stamping/forming is a strategic area which interests HEF. Coupling HEF's surface engineering technologies with innovative and emerging tool manufacturing technologies is a focus area for HEF. Therefore, partnering with ORNL gives HEF a strategic advantage to leverage some of the emerging technologies in this area. This partnership is critical for further expanding HEF's customer base in the United States and the United States manufacturing industry in general.

The work was conducted in two phases. In the phase 1 410SS was the material of focus. Based on the results of phase 1, the test matrix was updated for the remaining materials and phase 2 served to evaluate the efficacy of the surface engineering process other tool materials. In addition, the feasibility of using the proposed surface treatment/coating to enhance the performance of a component level sample were also studied.

2. MATERIALS AND EXPERIMENTS

2.1 MATERIALS, HEAT TREATMENT, AND NITROCARBURIZATION

Four materials, 410SS (ER410), 410NiMo SS (ER 410NiMo), 630 SS (ER630), and Maraging 250 (AMS 6512) steels, were printed by WAAM, which 410SS was printed and studied in phase 1 and the other three materials were printed and studied in phase 2.

In the phase 1 of this study, the first part consisted of depositing a wall of 410 SS using WAAM. The wall was 17 inches tall, 23 inches wide and 0.4 inches thick. Waterjet cutting and wire EDM were used to section the printed wall into 70 samples with the pattern shown in Figure 1, and materials within 1 in. from the top, bottom, and two side edges of the printed wall were discarded. After the sample section, all samples were machined to 50 × 50 × 5 mm dimensions. Out of the machined 70 samples, 35 samples (A1-1 – C2-5 inside the red frame in Figure 1.) were used to test 7 different conditions (combinations of temperature and time), using 5 samples from the same column for each test condition. The rest 35 samples were kept on reserve. The surface treatment process involves degreasing and rinsing, followed by drying and preheating the parts that are finally subjected to the ARCOR® process. This process is a nitrocarburizing process conducted in a fused cyanide-free salt bath.

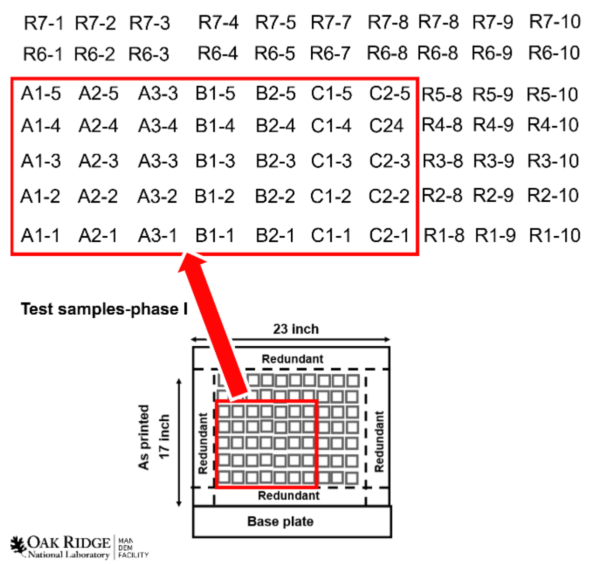


Figure 1. WAAM printed 410 SS wall sample arrangement plan.

For phase 2, two heat treatments were applied on some printed materials. Materials were machined to 50 × 50 × 5 mm coupons for nitrocarburization. The nitrocarburization processes were performed under a combination of three temperatures (950, 1075, and 1165 °F) and four different lengths of time (45, 60, 90, and 120 min). Five coupons were nitrocarburized in each material, heat treatment, and nitrocarburization condition. The summary of material, heat treatment and nitrocarburization conditions are shown in Table 1, where AC represents air cooling.

Table 1. Material, heat treatment, and nitrocarburization conditions.

<table>
<tr><th rowspan="2">Material</th><th rowspan="2">Status</th><th rowspan="2">Heat treatment</th><th colspan="7">Nitrocarburization</th></tr>
<tr><th colspan="2">950 °F</th><th colspan="3">1075 °F</th><th colspan="2">1165 °F</th></tr>
<tr><td>410 SS</td><td>As-printed</td><td>N/A</td><td>60 min</td><td>120 min</td><td>30 min</td><td>60 min</td><td>120 min</td><td>60 min</td><td>120 min</td></tr>
<tr><td>410NiMo</td><td>As-printed</td><td>N/A</td><td rowspan="5">90 min</td><td rowspan="5">120 min</td><td rowspan="5">60 min</td><td rowspan="5">90 min</td><td rowspan="5">120 min</td><td rowspan="5">45 min</td><td rowspan="5">90 min</td></tr>
<tr><td>630</td><td>As-printed</td><td>N/A</td></tr>
<tr><td>630 (17-4PH)</td><td>Heat treated</td><td>1900F/30min/AC</td></tr>
<tr><td>Maraging 250</td><td>As-printed</td><td>N/A</td></tr>
<tr><td>Maraging 250</td><td>Heat treated</td><td>2100F/1h/AC + 1900F/1h/AC</td></tr>
</table>

After nitrocarburization, metallographic specimens were extracted out from coupons, and they were polished and etched for hardness testing and structure and chemistry characterization.

2.2 STRUCTURE AND CHEMISTRY CHARACTERIZATION

Material microstructure and hardened layer thicknesses were characterized using an optical microscopy (OM). Chemical element distributions along straight lines from the specimen surface to the material substrate were detected by an electron microscopy (SEM) using energy dispersive X-ray spectroscopy (EDS).

2.3 HARDNESS TESTING

Rockwell C hardness tests were applied on nitrocarburized coupon surfaces for macro-hardness evaluation. Vickers microhardness tests were applied on nitrocarburized cross section specimens for local microhardness distributions.

2.4 MECHANICAL PROPERTIES TESTING

Tensile tests and Charpy impact tests were performed on WAAM printed materials of phase 2 at room temperature for strength, elongation, and fracture toughness evaluations. For each material and heat treatment conditions, three tensile tests were performed along the horizontal (welding torch moving) direction and vertical (building) direction, respectively. Prior to the mechanical property tests, heat treatment of 1075F/60min/AC was applied on all as printed and heat treated materials, simulating thermal effect during nitrocarburization.

2.5 STAMPING TOOL PRODUCTION AND CHARACTERIZATION

After testing and characterization, materials with the optimum post print heat treatment and nitrocarburization with the best parameters were selected for stamping tool manufacturing and processing. The tools were used for stamping cycles and a used tool was characterized and tested.

3. TECHNICAL RESULTS

3.1 STRUCTURE CHARACTERIZATIONS AND CHEMICAL ELEMENT DISTRIBUTIONS

After polishing and chemical etching, the oxide layer and nitrocarburized layer of 410NiMo stainless steel and 630 (17-4PH) stainless steel specimens were recognized under the OM, but the nitrocarburized layers of Maraging 250 steel were not clear under the OM. Near surface cross section images of a nitrocarburized 410NiMo specimen are shown in Figure 2, where the numbers with black background are measurements of nitrocarburized layer thickness and the number with blue background is the measurement of oxidated layer thickness.

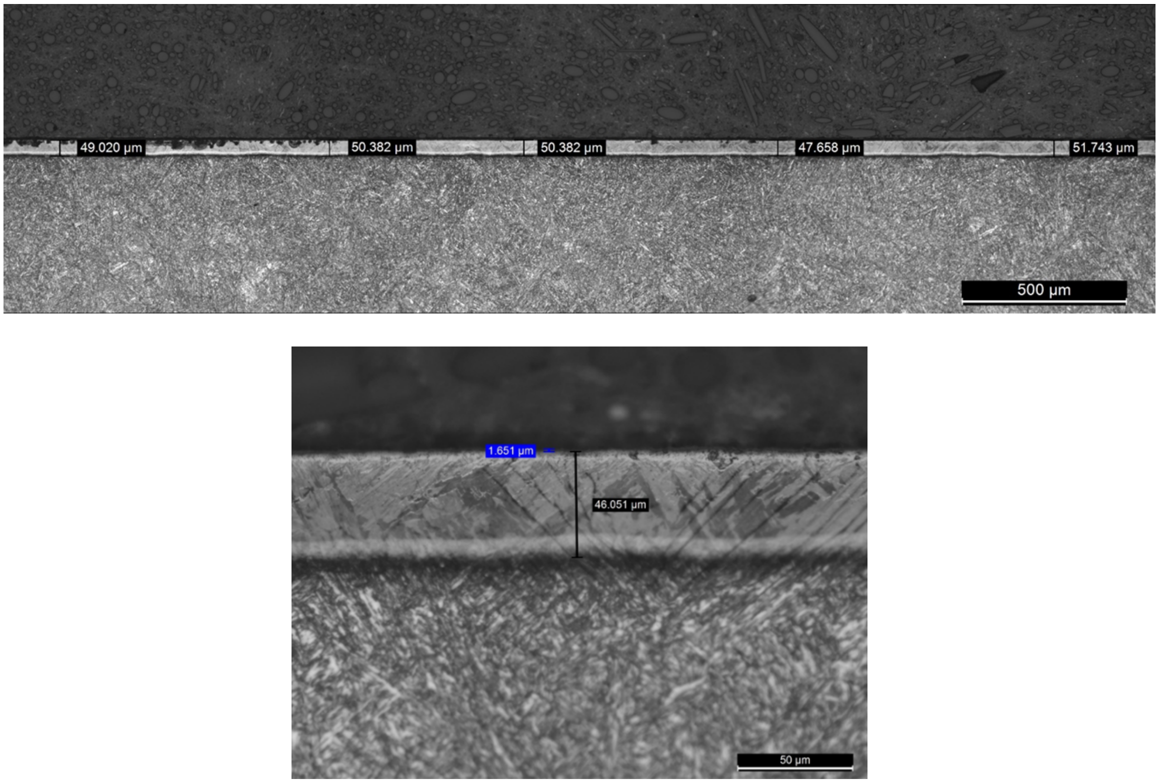
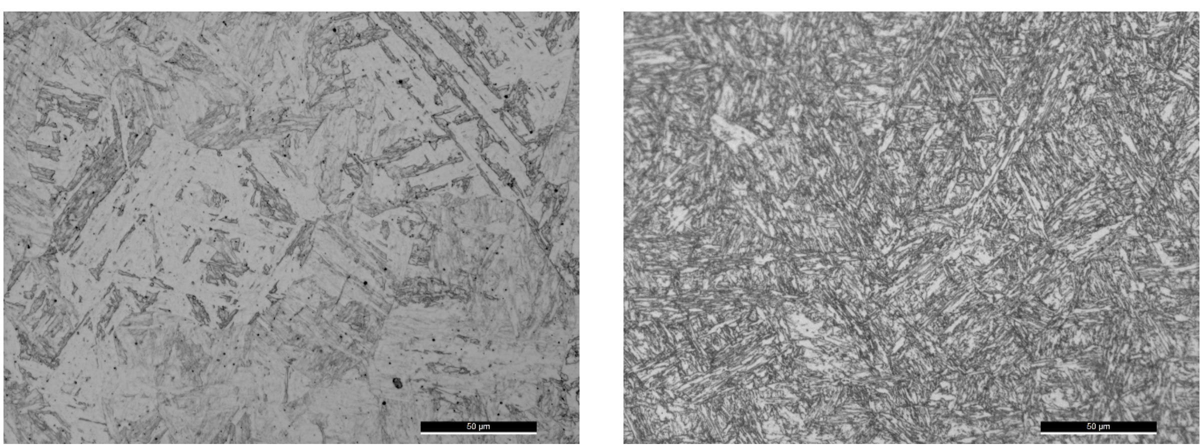
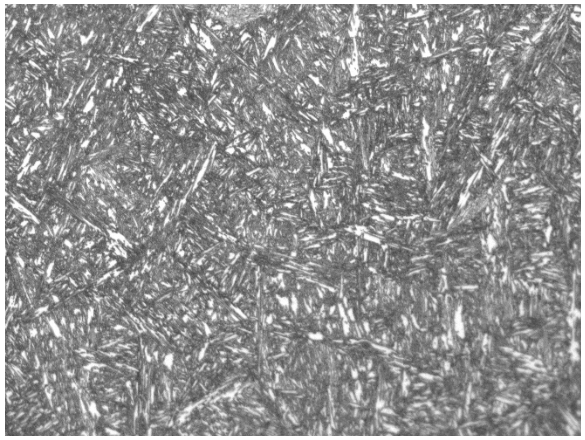


Figure 2. OM images of the oxidated layer and nitrocarburized layer after etching.

The elevated temperature in nitrocarburization affected material microstructures, similar to the tempering heat treatment effects. For example, 410NiMo SS microstructures after 90 min. nitrocarburization under different temperatures are shown in Figure 3.



(a) 950 °F　　(b) 1075 °F



(c) 1165 °F

Figure 3. 410NiMo SS microstructures after 90 min. of nitrocarburization at different temperatures.

With SEM/EDS, chemical element distributions close to the surface of selected specimens were obtained. Such chemical element changes distinguish the oxidated and nitrocarburized layers from the substrate. A set of SEM/EDS chemistry scanned results of a 410NiMo SS specimen are shown in Figure 4. The oxygen content was high in the oxidated layer (less than 10 µm thick from the surface) and the nitrogen content was high in the nitrocarburized layer (close to 50 µm thick).

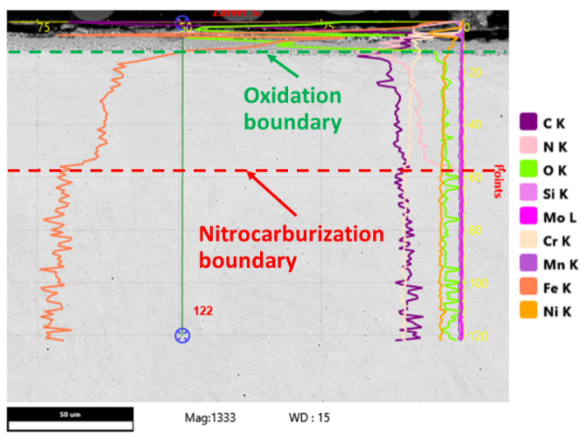


Figure 4. SEM/EDS line scan element distributions.

Combining the OM observation and the SEM/EDS scanning, material hardened layer thicknesses after nitrocarburization are shown in Table 2. From this table, the general trends are, 1, for the same material, the higher processing temperature and the longer processing time, the thicker the nitrocarburized layer, and 2, for the same nitrocarburization temperature and time, high alloyed material (Maraging 250 steel) resulted thinner nitrocarburized layer than those with relatively lower alloyed materials (410NiMo and 630 steels).

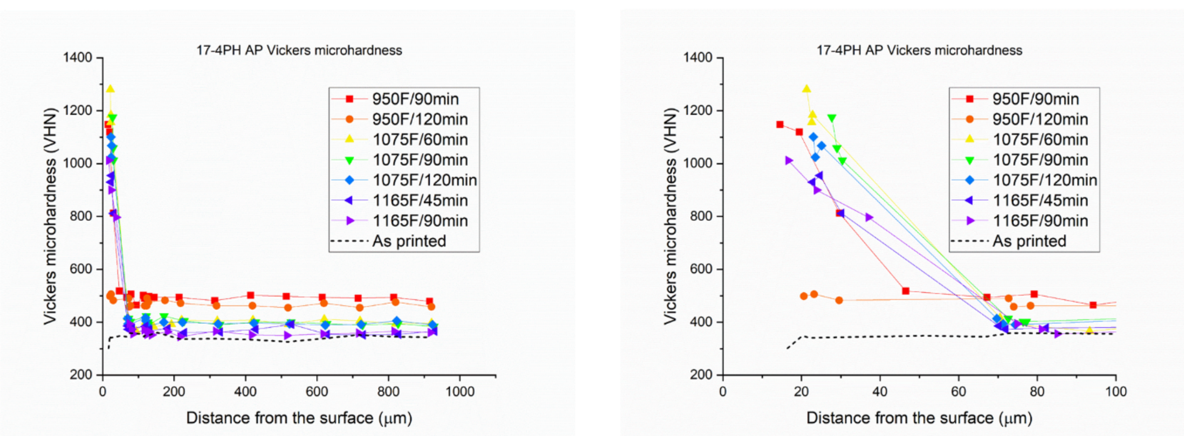
Table 2. Nitrocarburized layer thickness with different processing parameters.

Material	Status	Heat treatment	Nitrocarburization layer thickness, µm						
			950F 60min	950F 120min	1075F 30min	1075F 60min	1075F 120min	1165F 60min	1165F 120min
410 SS	As-printed	N/A	10	32	32	32	59	37	79

			950F 90min	950F 120min	1075F 60min	1075F 90min	1075F 120min	1165F 45min	1165F 90min
630	As-printed	N/A	17	20	23	43	46	42	50
630 (17-4PH)	Heat treated	1900F/30min/AC	12	16	35	41	45	47	55
Maraging 250	As-printed	N/A	6	11	N/A	N/A	35	31	N/A
Maraging 250	Heat treated	2100F/1h/AC + 1900F/1h/AC	4	8	N/A	N/A	31	34	N/A

3.2 HARDNESS MEASUREMENT

Vickers microhardness inside the nitrocarburized layers are much higher than those of the substrate. 410 SS showed average hardness of 1036 HV (±86) at 1075F, 620HV (±35) at 1165F and 562HV (±109) at950F. For 410NiMo SS and 630 SS, most nitrocarburization processing conditions resulted more than 1000 $HV_{0.1}$ (940 HV is equivalent to 68 HRC) in the hardened layer. The Vickers microhardness distributions of the as printed 630 SS are shown in Figure 5. As they are shown in Figure 5, the nitrocarburized layer hardness values are mainly between 1000 $HV_{0.1}$ to 1300 $HV_{0.1}$, while hardness of the substrates were between 300 – 500 $HV_{0.1}$, affected by the nitrocarburization thermal processes. Many low hardness values measured in nitrocarburized layers were artificial, i.e., caused by either the indentation was too close to the specimen surface and resulted large indentations, or the indenter partially indented in the substrate. The maximum measured microhardness in the Maraging 250 steel nitrocarburized layer was about 980 $HV_{0.1}$.



(a) Overall microhardness distributions (b) Microhardness close to surface

Figure 5. Vickers microhardness distribution of the as printed 630 stainless steel.

Surface Rockwell C hardness values were mainly between 25 – 50 HRC for all nitrocarburized material coupons. This is because the Rockwell C macro-hardness testing involved substrate materials and the measured value was dominated by the substrate hardness (50 HRC is equivalent to ~500 HV).

3.3 MECHANICAL PROPERTIES

Tensile properties and Charpy impact energies of WAAM printed materials are listed in Table 3. Each yield strength (YS), ultimate tensile strength (UTS), and elongation values showed in Table 3 are average values of six test results (three along the horizontal direction and three along the vertical direction) plus standard deviations. Elongations were measure manually after tensile tests. The as printed 410NiMo has the highest elongation and fracture toughness, and its strengths are relatively low. The heat treated 17-

4PH SS has higher yield strength and fracture toughness, and similar ultimate strength and elongation with the as printed condition. The Maraging 250 steels have high strengths, but the elongation and fracture toughness are relatively low for both as printed and heat treated conditions, and one attribution was voids or inclusions presented at some specimen fracture surfaces.

Table 3. Mechanical properties of WAAM printed materials.

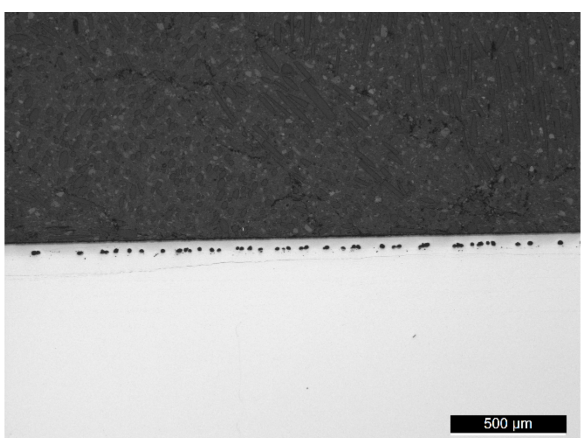
Material	Status	YS, ksi	UTS, ksi	Elongation, %	Charpy Energy, ft-lbs
410NiMo	As printed + aged	111±0.2	119±0.3	18.2±0.8	109
630	As printed + aged	135±10.4	164±0.5	15.4±2.0	22
630 (17-4PH)	Heat treated + aged	154±3.2	163±1.8	16.4±1.0	44
Maraging 250	As printed + aged	170±4.3	190±8.6	8.1±3.7	18
Maraging 250	Heat treated + aged	199±4.9	221±2.9	7.0±3.3	14

Aged: 1075F/60min/AC

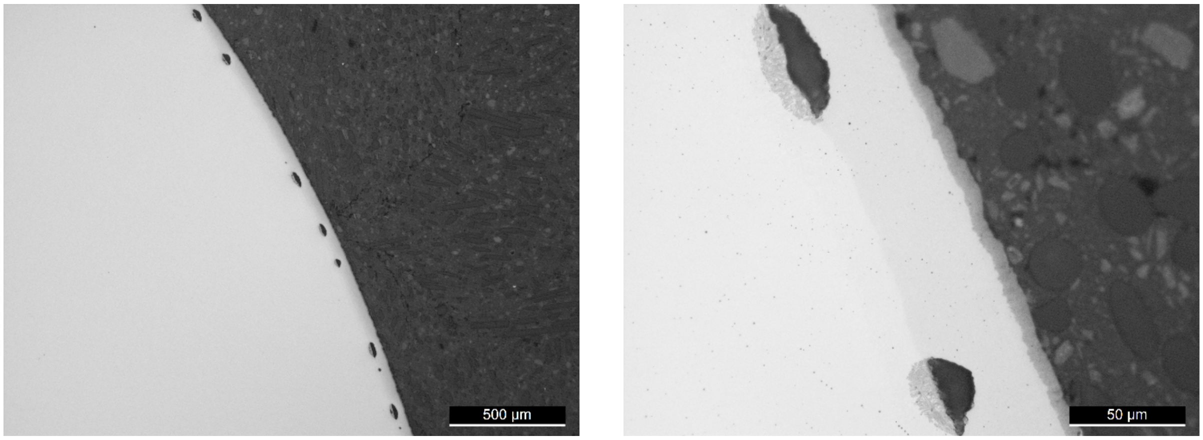
3.4 STAMPING TOOL PRODUCTION AND CHARACTERIZATION

Through the structure, hardness, and mechanical properties study, two materials, as printed 410NiMo and heat treated 17-4PH stainless steels, were chosen for stamping tool machining. The machined tools were nitrocarburized at 1075 °F for 120 min. Then the 17-4PH tool was used for 500 cycles of hot stamping without any lubricant. After that, the used tool was sent to ORNL for characterization.

Cross section images of specimens sliced out from the used stamping tool showed no crack or delamination developed inside the nitrocarburized layer and between the nitrocarburized layer and the substrate. However, void like features were observed at the interface of the nitrocarburized layer and the substrate, but there was no cracking initiated at the void/solid material corner. Images of the cross section with void features are shown in Figure 6. Further studies are needed to characterize these features and to identify the reason of their present.



(a) nonwork section



(b) Work section

Figure 6. Void features at the interface of the nitrocarburized layer and the substrate of the used 17-4PH stainless steel tool.

4. SUMMARY AND CONCLUSIONS

In summary, ORNL and HEF collaborated to develop defect-free nitrocarburized layers on 410SS, 410NiMo SS, 630 SS and Maraging 250 steel samples fabricated by wire arc manufacturing, with and without post print heat treatment. The WAAM printed materials exhibited good mechanical properties. The nitrocarburization generated extremely hard surface layer for all materials in every conditions. The hardened layer thickness increased with the increase of nitrocarburization temperature and time. The optimal heat treatment condition, nitrocarburization time and temperature of each material were identified. Stamping tools were fabricated using 410NiMo SS and 17-4PH SS, and then they were nitrocarburized with the selected optimization conditions. The 17-4PH steel nitrocarburized tool completed 500 cycles of hot stamping, and no crack or delamination was observed at the nitrocarburized cross sections. However, voids were observed at the nitrocarburized layer and substrate interface, and further study is needed to reveal the cause and find ways to eliminate these voids. Furthermore, nitrocarburized stamping tool life with more working cycles needs to be further verified.

5. ACKNOWLEDGEMENTS

The wire-arc technology used to manufacture the components was developed in collaboration with Lincoln Electric under a Cooperative Research and Development Agreement. The stamping 3D printed tools were machined and tested by Dienamic Tooling Systems (DTS).