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Laser Marking and Composition Analysis of 13-8 Steel and Nitronics 60 Surfaces

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Abstract

Pulsed laser irradiation is used to irradiate and mark 13-8 steel and Nitronics 60 parts in order to create observable markings on the surfaces. The best optical contrast ratio between marked regions and unmarked regions is desired for digital image correlation. The contrast is optimized by using pulsed-laser irradiation and varying the laser power, pulse length, and scan speed. X-ray diffraction was used to characterize the laser-irradiated surface, and it was found that oxide formation and surface roughness are responsible for the observed contrast.

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FIGURES

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NOMENCLATURE

CW	Continuous Wave
Fe	Iron
GIXRD	Grazing Incidence X-ray Diffraction
O	Oxygen
Ra	Average Surface Roughness

1. INTRODUCTION

We performed experiments to determine the pulsed-laser parameters necessary to form high contrast features on 13-8 steel and Nitronics 60 surfaces. Laser irradiation is often used to rapidly mark metallic surfaces with little modification to the bulk part. Pulsed laser light is used in our studies since the pulse intensity is sufficiently high to consistently modify the material surface, regardless of initial surface conditions such as roughness and contamination. Therefore, less pre-treatment of parts is necessary before laser irradiation. The pulsed nature of the laser light also means the thermal quench rates are higher, as compared to continuous wave (CW) irradiation, so the bulk part will not be significantly affected by heating during and after irradiation.

1.1. Experimental Setup

Table 1 lists the marking parameters for the SPI Lasers LLC, pulsed fiber laser selected for experiments, and Figure 1 shows the laser scan patterns used for marking.

Table 1. SPI Lasers, pulsed fiber laser parameters for marking Nitronics 60 and 13-8 steel.

Beam Shape	Gaussian
$1/e^2$ Focused Beam Diameter	60 µm
Focal Length	18.5 cm (7.3")
Repetition Rate	60 kHz
Wavelength	1064 nm
Hatch Spacing	10 µm

The laser was focused onto the stationary part and scanned at a constant scan speed using galvanometric mirrors. All experiments were performed in air. The laser beam scanned horizontally (red arrows) across the part and turned off at the end of each row in order to maintain a constant scan speed over the marked regions, outlined with dashes in Figure 1. The laser scans over a given region using one pass.

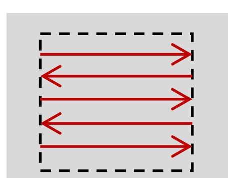

Figure 1. Patterns for laser scanning over marked regions of Nitronics 60 and 13-8 steel. The red arrows indicate the laser scan direction and the distance between rows (hatch) is 10 µm. The laser turns off at the end of each row.

2. RESULTS

2.1. Contrast Analysis

Figure 2 shows bright-field optical microscopy images of example test patterns made using a 15 ns pulse length, an average power of 3 W, and a 20 mm/s scan speed.

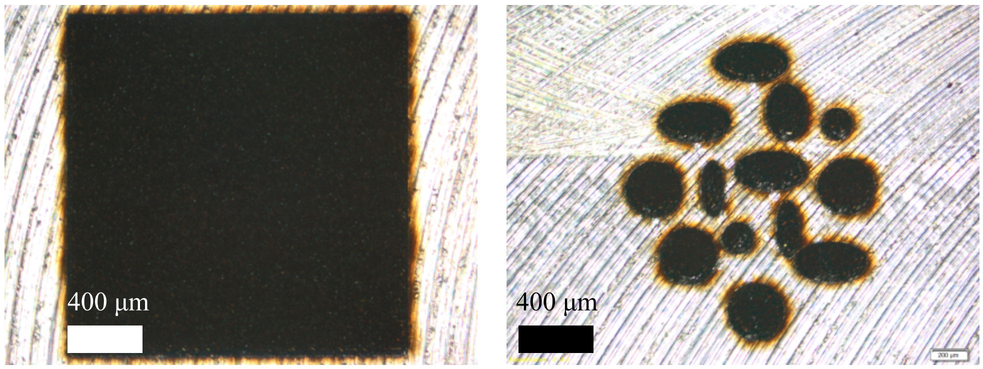

Figure 2. Test patterns marked on the surface of Nitronics 60 using a 15 ns pulse length, 3W average power, and 20 mm/s scan speed.

The dark contrast samples, such as those in Figure 2, may have some re-deposited material on the surface; however, post-treatment cleaning of the sample in Figure 2 has shown that the re-deposited debris is not easily removed from the surface. For additional experiments involving scan speed variation, the laser pulse length, power, and repetition rate were held constant. Varying the scan speed provided access to different accumulated laser fluences; where the accumulated fluence is the total J/cm^2 delivered to a given area by laser irradiation. Typically, higher accumulated laser fluences lead to more surface modification.

Figure 3 shows bright-field optical microscopy images of test patterns on Nitronics 60 obtained using a 20 ns pulse length, a 3 W average power, a 60 kHz repetition rate, and a 10 μm hatch spacing. The scan speeds (mm/s) starting at the left, top row and moving right and down are: 5000, 4500, 4000, 3500, 3000, 2500, 2000, 1750, 1500, 1250, 1000, 900, 800, 700, 600, 500, 450, 400, 375, 350, 325, 300, 290, 280, 270, 260, 250, 240, 230, 220, 210, 200, 190, 180, 170, 160, 150, 140, 130, 120, 110, 100, 90, 80, 70, 60, 50, 40, 30, 20, and 10 mm/s. It can be seen that higher scan speeds correspond to lighter contrast irradiated regions.

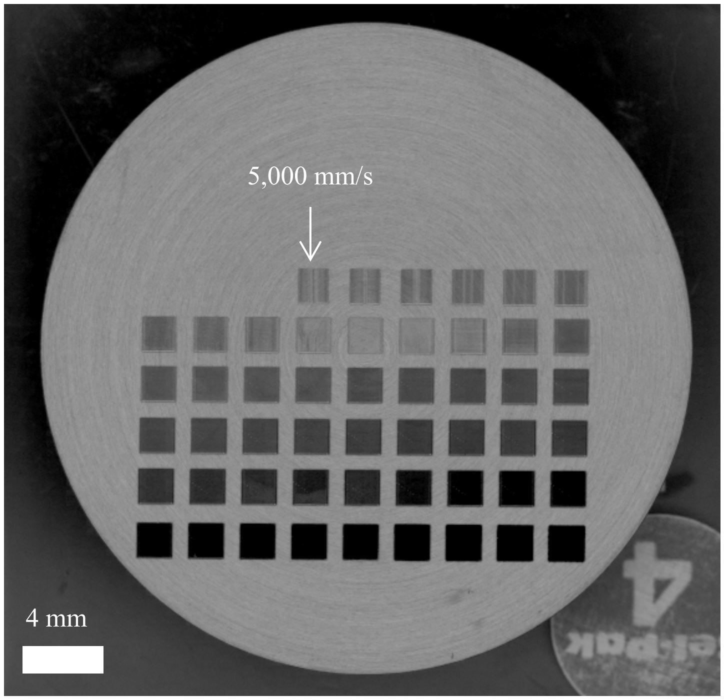

Figure 3. Test patterns made in Nitronics 60 using a 20 ns pulse length, 3 W average power, 60 kHz repetition rate, and increasing scan speed from left-right and top-bottom.

Figure 4 shows a 13-8 surface marked using the same laser conditions as the Nitronics 60 sample.

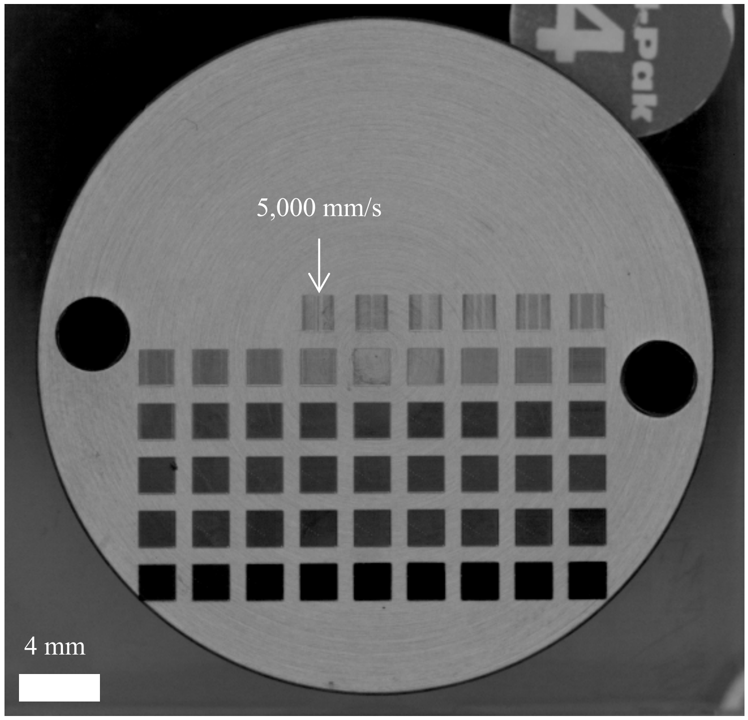

Figure 4. Test patterns marked in 13-8 steel using a 20 ns pulse length, 3 W average power, 60 kHz repetition rate, and increasing scan speed from left-right and top-bottom.

The samples were measured using a light dome for illumination and recording with a Phantom 1610 12-bit high-speed camera at different in-plane, sample rotation angles (0°, 45°, 90°, 135°, 180°, 225°, 270°, and 315°). The average reflected intensity of square regions (11 x 11 pixels) from bright, non-marked surfaces were measured and subtracted from the average reflected intensity of square regions (11 x 11 pixels) marked by the laser. The difference between counts (Δ) was averaged over the 8 rotation angles. The percentage deviation (%) is calculated from the ratio between Δ and the maximum possible count (4095). The highest scan speeds did not always have consistent reflectance measurements within a given irradiation area, in these cases the counts are not listed. The results for Nitronics 60 are shown in Table 2.

Table 2. Contrast analysis for Nitronics 60 laser marked regions.

Scan Speed (mm/s)	Δ	%
Original Surface	2311	
10 - 70	2311	56.4
80	2310	56.4
90	2311	56.4
100	2307	56.3
110	2306	56.3
120	2294	56.0
130	2140	52.3
140	1658	40.5
150	1823	44.5

160	1510	36.9
170	1613	39.4
180	1505	36.8
190	1438	35.1
200	1374	33.6
210	1390	33.9
220	1402	34.2
230	1328	32.4
240	1276	31.2
250	1236	30.2
260	1216	29.7
270	1216	29.7
280	1238	30.2
290	1181	28.9
300	1183	28.9
325	1138	27.8
350	1104	27.0
375	1009	24.6
400	994	24.3
450	975	23.8
500	996	24.3
600	1012	24.7

The results obtained from laser marked 13-8 are shown in Table 3.

Table 3. Contrast analysis for 13-8 laser marked regions.

Scan Speed (mm/s)	Δ	%
Original Surface	2176	
10 - 60	2176	53.1
70	2174	53.1
80	2168	52.9
90	2169	52.9
100	1745	42.6
110	1582	38.6
120	1456	35.5
130	1401	34.2
140	1430	34.9
150	1627	39.7
160	1579	38.5
170	1452	35.5
180	1503	36.7
190	1365	33.3
200	1356	33.1
210	1350	33.0

220	1397	34.1
230	1387	33.9
240	1426	34.8
250	1368	33.4
260	1380	33.7
270	1382	33.7
280	1424	34.8
290	1402	34.2
300	1427	34.8
325	1484	36.2
350	1344	32.8
375	1356	33.1
400	1382	33.7
450	1345	32.8
500	1275	31.1
600	926	22.6
700	686	16.8
800	566	13.8
1250	533	13.0
1500	764	18.7
1750	861	21.0
2000	833	20.3

The surface roughness of the laser marked samples was measured using a Dektak profilometer employing a 5 micron radius tip. The average surface roughness (Ra) for the original 13-8 surface was measured to be ~ 380 nm. For the original Nitronics 60 surface, Ra ~ 260 nm. For 13-8, the surface roughness did not change significantly after laser irradiation for scan speeds 80 – 5,000 mm/s. For scan speeds 60 and 70 mm/s, the Ra increased to ~ 500 nm, and for 10 – 50 mm/s, the surface roughness increased to a Ra ~ 5 µm. For Nitronics 60, the Ra did not significantly change for scan speeds 140 – 5,000 mm/s. The Ra increased to ~ 500 nm for scan speeds 60 – 130 mm/s. For scan speeds 10 – 50 mm/s, the Ra varied between 1 and 5 µm.

2.1. Grazing Incidence X-ray Diffraction

Grazing incidence x-ray diffraction (GIXRD) was used to identify phases which formed after laser irradiation. GIXRD patterns for Nitronics 60 are shown in Figure 5. The scan speeds used to make these features were as follows: region 1 = 400 mm/s, region 2 = 2000 mm/s, region 3 = 120 mm/s, and region 4 = 10 mm/s. Austenite was observed for all four laser irradiated regions, and region 4 showed evidence for Fe_3O_4 spinel phase formation. Regions 1 – 3 show evidence for a martensite phase, which is also likely present prior to laser irradiation.

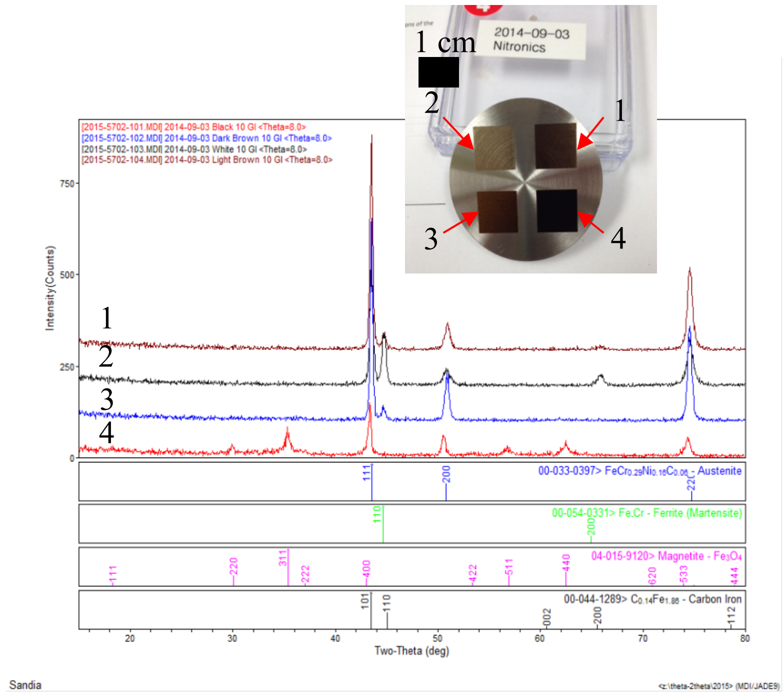

Figure 5. GIXRD analysis of Nitronics 60 after laser irradiation at four scan speeds: 10, 120, 400, and 2000 mm/s.

GIXRD patterns for 13-8 steel are shown in Figure 6. The laser scan speeds used to make these features were as follows: region 1 = 220 mm/s, region 2 = 350 mm/s, region 3 = 120 mm/s, and region 4 = 10 mm/s. Austenite was only observed for region 3. Region 3 also showed evidence for Fe_3O_4. Regions 1,2, and 4 showed evidence for martensite.

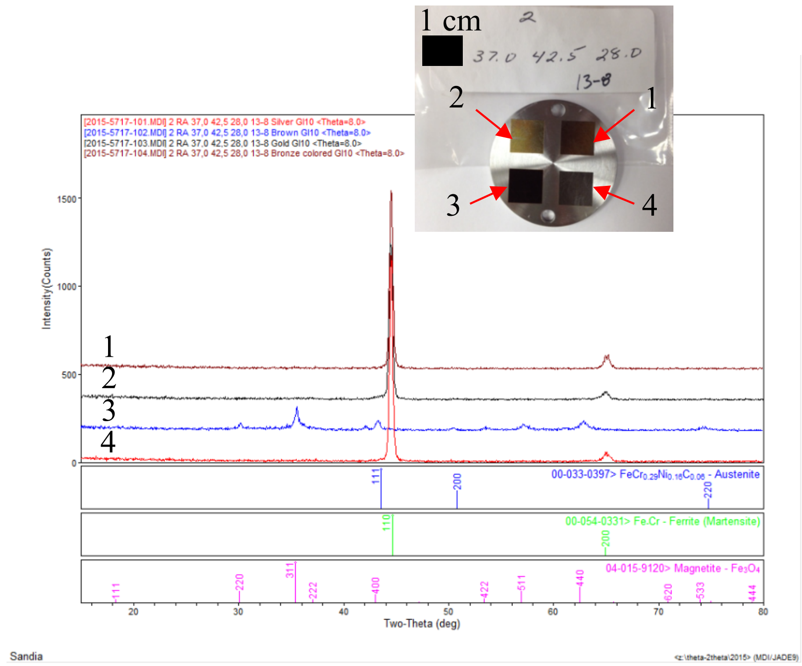

Figure 6. GIXRD analysis of 13-8 steel after laser irradiation at four scan speeds 10, 120, 220, and 350 mm/s.

3. DISCUSSION

Taking into account the fact that the Ra did not significantly change due to laser irradiation until the scan speed was decreased to 50 mm/s, it is likely that the darker contrast seen for lower scan speed samples is due to both surface roughness and oxide formation. The significant increase in surface roughness may be the result of material removal and redeposition onto the irradiated surface. It is also apparent that, for the 13-8 steel, lower scan speeds (higher accumulated fluences) form austenite, while higher speeds do not show evidence for austenite.

For higher scan speed samples, the surface roughness did not significantly increase after laser irradiation. Therefore, the contrast changes for higher scan speed samples, as compared to the original 13-8 and Nitronics 60 surfaces, are mostly due to oxide formation.

3.1. Extension of Findings to Rofin PowerLine E Series Laser

These finding have been applied to a Rofin PowerLine E series laser marker. Consistent marking of Nitronics 60 using this laser was desired, but there were problems when using continuous wave CW laser irradiation. It is thought that the CW beam did not have a sufficient laser intensity to mark surfaces under varying conditions such as dirty or rough surfaces. We operated the laser in pulsed mode, using a 1 μs laser pulse, and successfully achieved dark surface markings on both smooth and rough metallic surfaces. The best results were achieved by using a 1 μm step overlap, 60 kHz repetition rate, 28 amps, and allowing 100 pulses to irradiate the surface at each step. When using these settings, the laser can mark a 1 x 1 mm area in < 1 minute. The pulsed laser marks both smooth and rough surfaces more consistently because the laser intensity is much higher, as compared to CW laser light. The higher laser intensity prevents reflectivity differences due to surface roughness from significantly changing the laser-material interaction mechanisms.

4. CONCLUSIONS

In conclusion, we recommend that laser marking of 13-8 and Nitronics 60 parts should always be performed in pulsed mode. Even though the darkest contrasts appear to have some re-deposited material, post-treatment cleaning has shown that the re-deposited debris is not easily removed from the surface. The high laser intensities in pulsed mode allow for material removal and surface oxidation, even for rough surfaces; therefore, less pre-treatment of the surface is needed before laser processing is performed.

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