



Characterizations of Laser Glazed Surfaces and Substrate Structures of Nickel Alloy 718 for Engineering Applications

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ABSTRACT

Characterizations of laser glazed surfaces and substrate structures of nickel alloy 718 have been studied. Cross sections of laser glazed and substrate sample surfaces of nickel alloy 718 were cut to different dimensions for various tests. Morphological examinations of the sample surfaces were done using confocal laser scanning microscope. It was followed with x-ray diffraction (XRD) tests at different angles of incidents for identification of possible oxide phases in the alloy. Microstructural examinations of the alloy samples were carried out and they involved; scanning electron microscopy (SEM), electron back scatter diffraction (EBSD) and energy dispersive x-ray spectroscopy (EDX). The results of the tests from the sample morphologies show that the surfaces of the samples were covered with different oxide layers/scales. XRD results identified these oxide phases present on the sample surfaces with their various crystal systems. The results further show that as the angle of x-ray incident increased, the intensity of substrate phases increased with corresponding decrease in surface oxide phases. The results of electron back scatter diffraction from the band contrast map show that the microstructures of the laser glazed surfaces are columnar while that of the substrates are bimodal with small equiaxed grains surrounding the large grains. There was no evidence of distinct interfacial separation seen between the surfaces. The result further revealed different crystallographic plane orientations from Euler colour map and the possible slip planes corresponding to $\langle 001 \rangle$, $\langle 111 \rangle$ and $\langle 101 \rangle$. From the crystallographic colour orientations, it indicated that both the substrate and laser glazed regions are partly homogeneous. Results from scanning electron microscopy (SEM) of the sample substrate revealed the microstructures and the presence of bright spot believed to be intermetallic phases of heavy metals. Consequent upon these, analysis of both surfaces show that they have similar physical and microstructural properties with no interfacial separation at their boundary regions. These indicate that both structures are compatible for engineering applications.

Keywords: Energy Dispersive X-ray Spectroscopy (EDX); X-ray Diffraction (XRD) Tests; Substrate Structures; Laser Glazed Surfaces; Nickel Alloy 718

1. Introduction

Alloy 718 is a corrosion-resistant Ni-Cr-Fe alloy developed for use where high strength and corrosion resistant applications are highly required. These properties have seen the wide usage of this alloy in oil and gas sectors. Other areas considered to be of potential material use of this alloy are in nuclear power plants, gas turbines, aircraft engines and generally where strength and corrosion resistance are considered most in engineering applications (Deleume, Cloué, Andrieu and Devaux, 2008). This alloy can be used in the low strength (non-aged) or high strength (precipitation-hardened) with high creep stress rupture strength condition and it possess good weld ability without post welding crack (Kimmerl, 2014). Amongst the super alloys, Ni-based super alloy is normally regarded as the king or workhorse of super alloys because 50% of the weight of an aero-engine turbine is made of the alloy (Ede and Onah, 2021).

Precipitation-strengthened nickel-based super alloys are extensively utilized in the important industrial applications such as land-based and aero gas turbines due to their excellent high temperature strength (Tian, 2014). The process of coating hard phase particles on the surfaces of ductile matrix material with the aim of producing wear resistant surfaces is called laser glazing. In this method, hard particles like carbides, borides or silicide with more ductile materials are heated and subjected towards a substrate by plasma. In laser glazing surface technology, the hard particles and the ductile matrix are normally subjected to high temperature condition with the aim to dissolve them on the surface to produce better surfaces. A better adhesive bond with less porosity is achieved in this method, though the entire process is noisy and expensive. This technology works where the hard particles must be in equilibrium with the ductile substrate material and thick layer is required. The temperature required from laser is so high that volatilization of the powder will propel it to the substrate and cause it to be deposited on the substrate. This is applicable to deposition of material that has high vapor pressure at a high temperature above the melting point of the coating material (Schaefer, Ayers and Tucker, & 1981).

Figure 1 is a schematic image of laser glazed surface and the substrates.

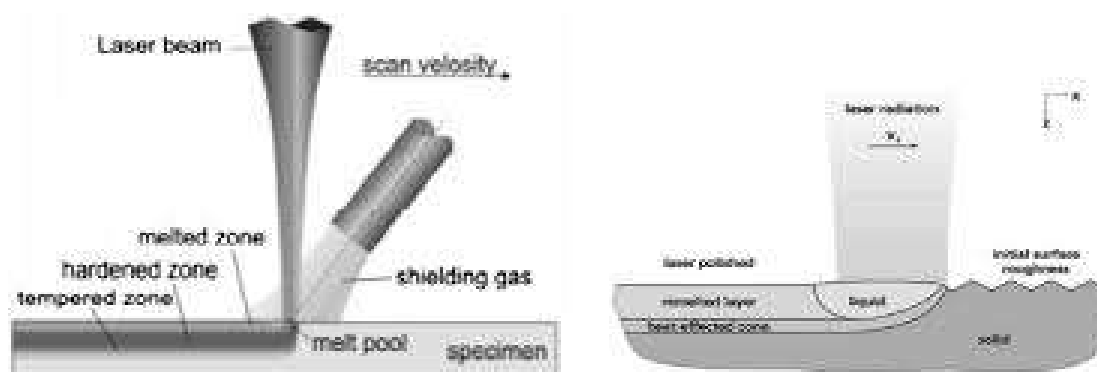


Figure 1: Schematic image of laser glazed surface and the substrate (Madhav, 1999)

Full characterization of laser surfaces together with the substrate structure upon which it is embedded on is very important in the engineering applications of this material. However, many researchers have worked in related areas of this field and came out with some important findings. Zhao et al (2009) studied the effects of heat treatment on microstructure and mechanical properties of laser solid forming of Inconel 718 alloy and discovered that the laser treatment resulted in microstructure of columnar dendrites, which grew epitaxial from the substrate (Zhao et al, 2009). Laser heat treatment of HVOF coated Inconel 718 alloy was studied by Cho et al. They demonstrated that the laser-heating improved the surface properties of Inconel 718 alloy considerably (Cho et al., 2010). Liu et al investigated the influence of laser scanning path on microstructures and mechanical properties of the laser solid formed Inconel 718 alloy and found out that the continuous directional growth of columnar grains was inhibited and the orientation deviation of dendrites in two adjacent layers increased after the laser treatment process (Liu et al, 2011). Consequent upon these, the aim of this work is to carry out comprehensive studies on the macro and microscopic analysis of both the laser glazed and substrate surfaces for engineering applications. The ways to achieve these are by the use of some metallurgical analytical methods like confocal laser scanning microscopy, x-ray diffraction (XRD), scanning electron microscopy (SEM), electron back scatter diffraction (EBSD) and energy dispersive x-ray spectroscopy (EDX) (Yilbas, Al-Aqeeli, and Karatas, 2016).

2. Experimental Methods

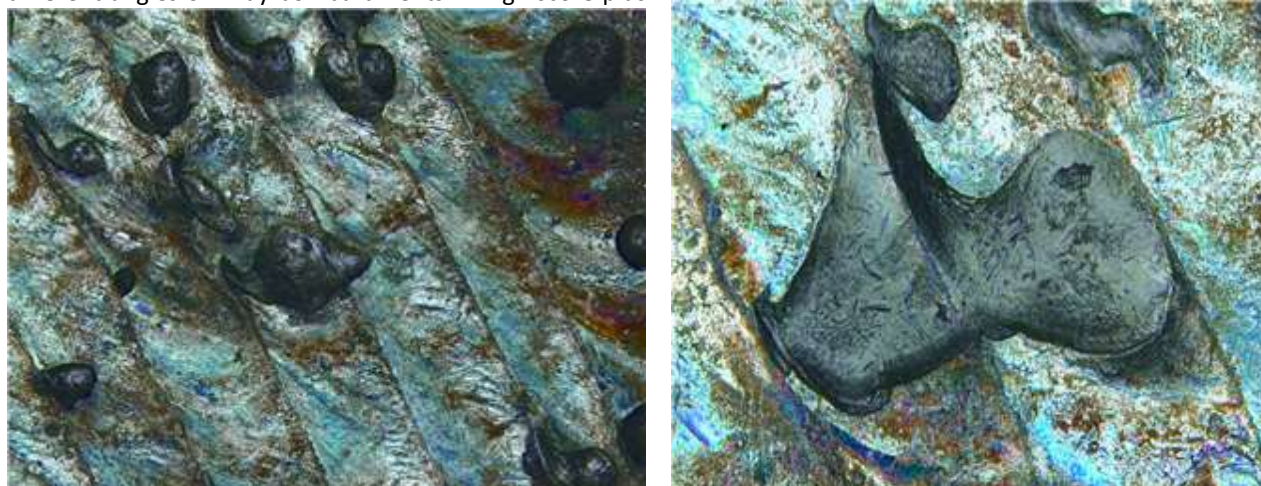
Various experimental tests were carried out and the tests involved laser scanning confocal microscopy, x-ray diffraction (XRD), microstructural analysis which involved optical microscopy, scanning electron microscopy (SEM), electron diffraction x-ray spectroscopy (EDX) and electron back scatter diffraction (EBSD). In laser scanning confocal, samples morphologies were examined with the confocal microscope. This was done by focusing a beam of light from laser through an aperture with the aid of an objective lens on the sample to be examined and images were built up pixel by pixel by gathering the emitted photons from the flourophores in the sample (Dalio, 2017) XRD technique was used to analyze alloy 718 sample to identify phases present in the material. Here the sample was placed in the x-ray analyzing chamber, gradually rotated while x-ray was bombarded on the material. During the analysis, when an x-ray incident on the alloy sample a diffraction pattern was created. The results of diffraction pattern collected were cross matched with thousands of diffraction patterns present in the storage library and the possible phases present in the sample were identified (Patil, 2011). Microstructural analysis involved cutting, grinding, polishing and microstructural examination of the material's internal structure with the aid of optical and scanning electron microscopes. It was followed by electron back scatter diffraction (EBSD). The technique gave information on strains, crystal orientation, phases and grain boundary mapping of the alloy material. EBSD analysis was done in scanning electron microscopy chamber where polished sample surface to be analyzed was tilted to an angle of approximately 70° to the diffraction camera so that the contrast in the electron back scatter diffraction pattern will increase to the required standard (Schwartz, Kumar, Adams, and Field, 2009) Finally, electron diffraction x-ray spectroscopy (EDX) analytical technique was used to determine the elemental compositions of material. The process works by the interaction between the sample and the x-ray source and the characterization was done on the basic principle that an element has a unique atomic structure that allows a unique set of peaks on its electromagnetic emission spectrum. To activate the emission of x-ray from a sample, a high-energy beam of excited particles like electrons, photons or beam of x-rays were concentrated on the sample. In this process, the focused incident beam excited an electron from the inner shell, ejected it from the shell and corresponding electron hole was created from the position of the electron. Consequent upon this, an electron from the outer shell with higher energy level occupied the hole and difference in the energy level was emitted in the form of x-ray and measured by energy dispersive spectrometer (Goldstein, 2012)

3. Results and Discussions

The results of laser scanning confocal microscopy images are as shown in figure 2. The images examined the morphology of the laser glazed surfaces of the material. Close looks at the samples show that the surfaces of the material were covered with different oxide films/scales. In some areas on the surfaces, film defects were formed on the surfaces during laser glazing. The laser optical micrographs also revealed that the surfaces were not smooth; it has a lot of surface variations and parallel contour lines across the surfaces. These phenomena were as a result of high temperature corrosion (oxidation) during laser forming process. The alloy was subjected to high temperature during laser glazing which led to oxidation and formation of different oxide layers on the surfaces.

Figure 2: Laser morphology surface image of alloy 718 sample

Table 1 shows the list of oxide phases and their crystal systems present on the surfaces of glazed alloy 718 at different angles of x-ray bombardments. A high score plus



software (version 4.8 October 2018) was used and a total of thirteen different oxide phases with five crystal systems were identified. Some of these oxides appeared repeatedly in different peaks within a diffraction pattern of the x-ray. Figures 3, 4, 5, and 6 show the diffraction patterns of peaks at 2° , 3° , 6° , and 9° angles of incidents respectively. It was observed that at small angle of incident, the substrate material has low signal while the oxide layers have high signal from the diffraction of the peak's intensity. As the incident angle increased, the substrate signal became stronger. The XRD showed all the oxides present in the material we are investigating and it is believed that the presence of these oxides has contributed to the physical and chemical properties of this material significantly. From the pictures of laser scanning confocal microscopy bulk of these oxides were found on the surfaces of the sample in line with the results of x-ray diffraction pattern that identified strong signal of the oxides at small angle of incident.

Table 1: List of oxide phases and their crystal systems

S/No	Oxide phases	Crystal Systems
1	Iron oxide	Cubic
2	Aluminium oxide	Cubic
3	Chromium oxide	Rhombohedral
4	Titanium oxide	Orthorhombic
5	Titanium oxide	Monoclinic
6	Molybdenum oxide	Cubic
7	Aluminium iron nickel	Cubic
8	Nickel titanium	Cubic
9	Aluminium oxide	Rhombohedral
10	Niobium oxide	Monoclinic
11	Aluminium molybdenum nickel	Cubic
12	Titanium oxide	Tetragonal
13	Iron oxide	Orthorhombic

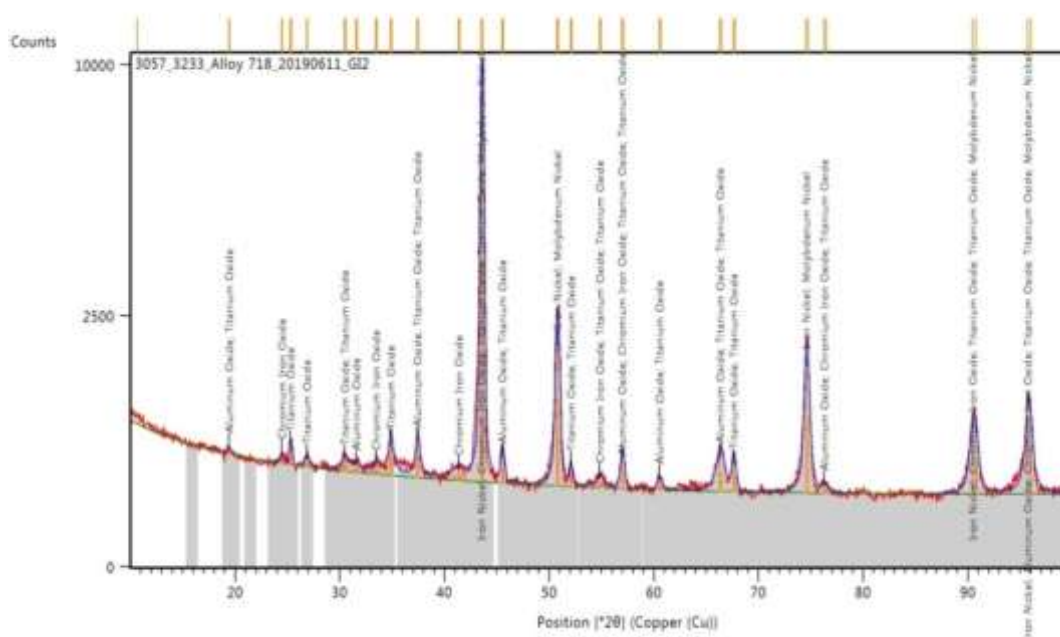


Figure 3: X ray diffraction pattern at 2° incident angle

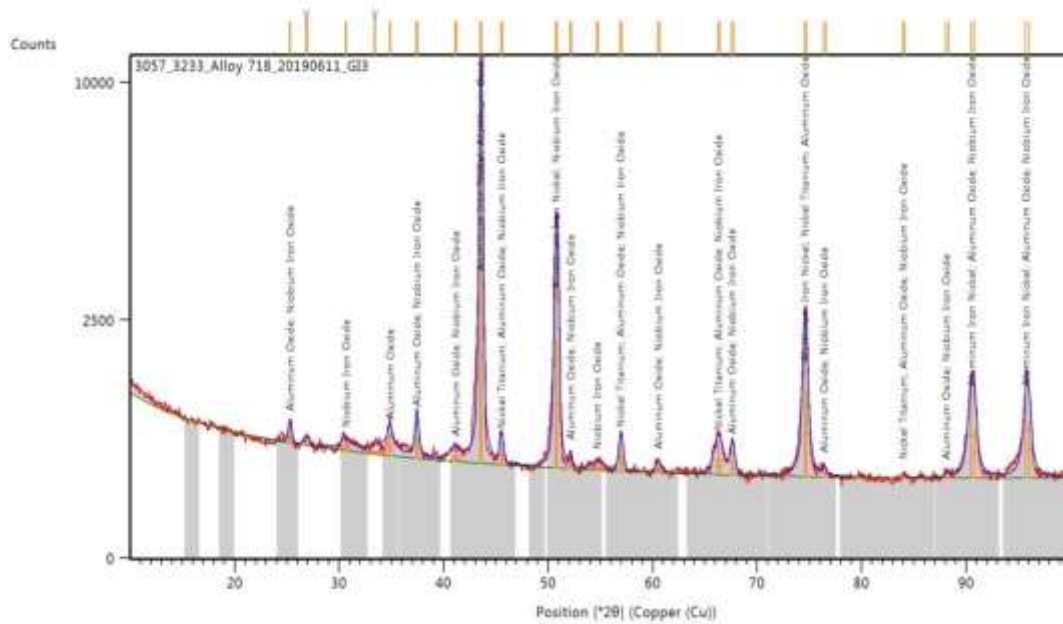


Figure 4: X ray diffraction pattern at 3° incident angle

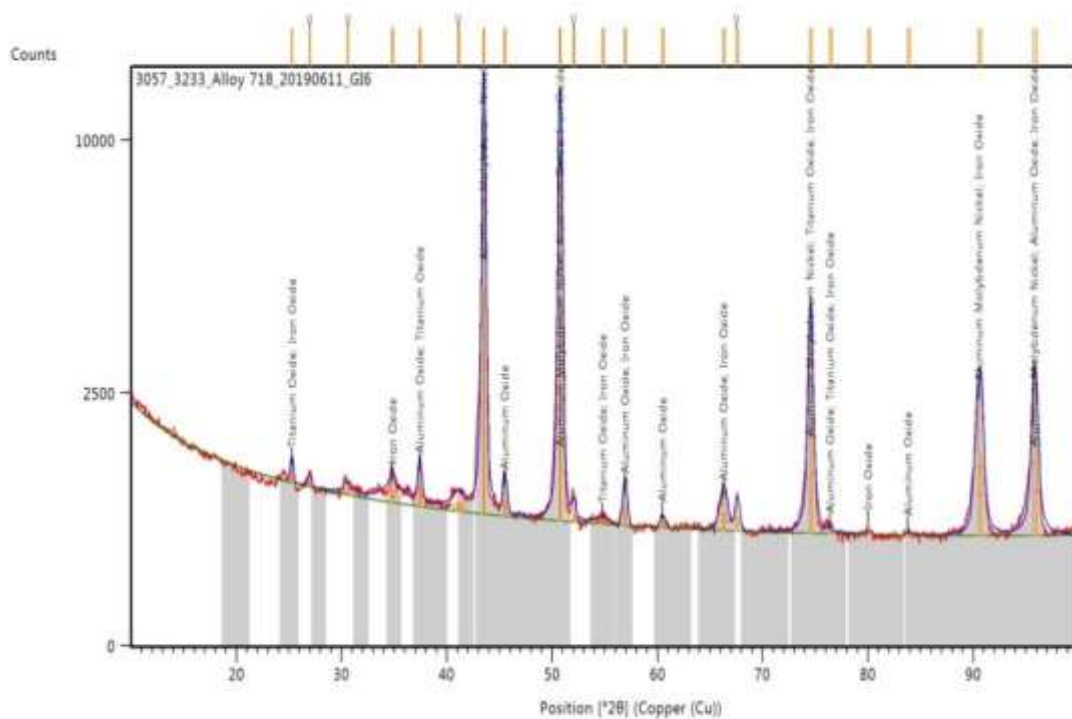


Figure 5: X ray diffraction pattern at 6° incident angle

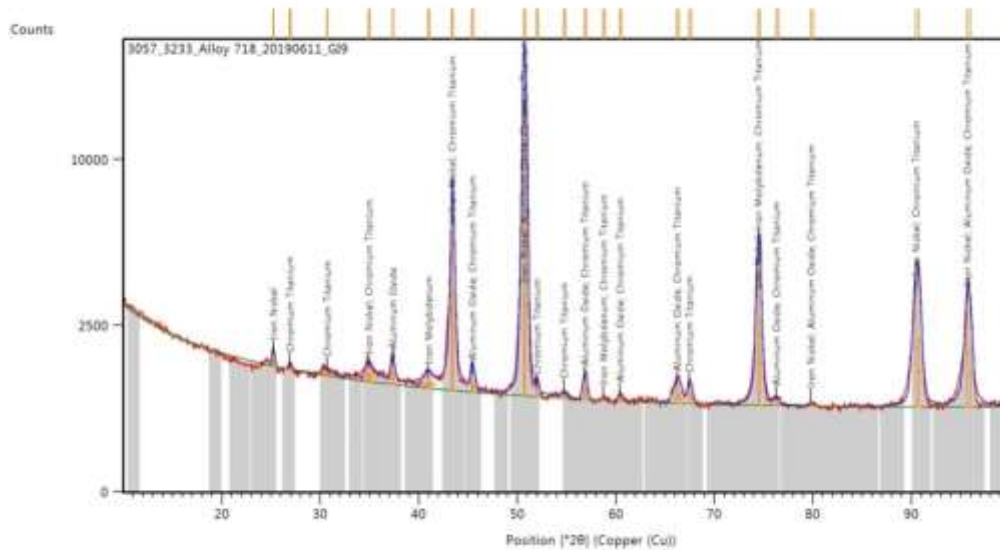


Figure 6: X ray diffraction pattern at 9° incident angle

The micrographs of band contrast map and Euler colour map orientation from electron back scatter diffraction (EBSD) are shown in figures 7 and 8. The region at the top most part of the micrograph represents the laser glazed area. A close look at the grain structures shows that the region has columnar microstructures that run vertically from the substrate material to the top. The microstructure of the substrate material is bimodal, it consists of large grains surrounded by small grains that are equiaxed in nature and this shows that substrate region has duplex structure. The Euler colour map in figure 8 shows partly homogenous plane orientations in laser glazed and substrate regions. This is an indication that both regions have almost the same plane orientations. This columnar structure of the laser glazed region as observed, is in agreement with similar works done by some scholars. Zhao et al studied the effect of heat treatment on microstructure and mechanical properties of laser solid forming of Inconel 718 alloy.



Figure 7: Band contrast map of EBSD for substrate and laser regions

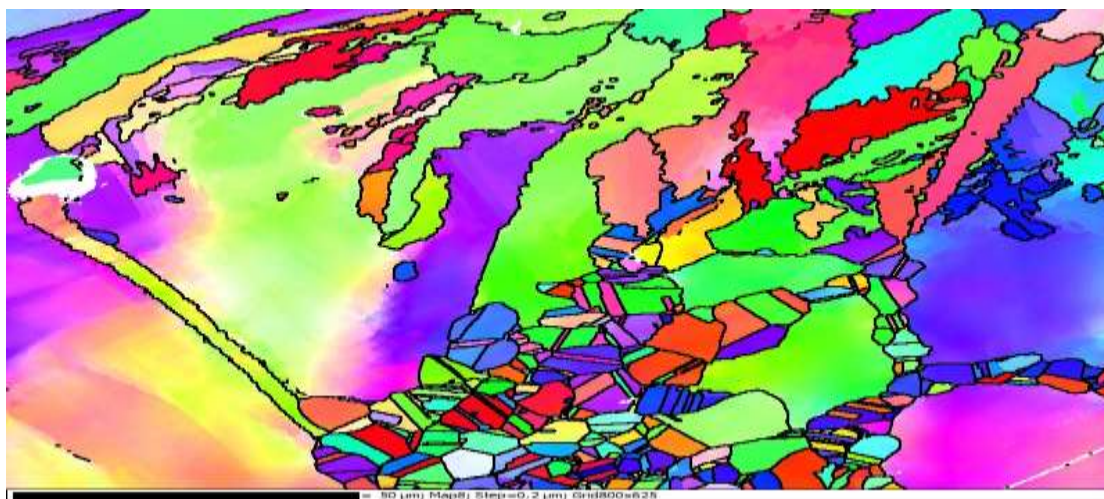


Figure 8: Euler colour map orientation of EBSD for substrate and laser regions

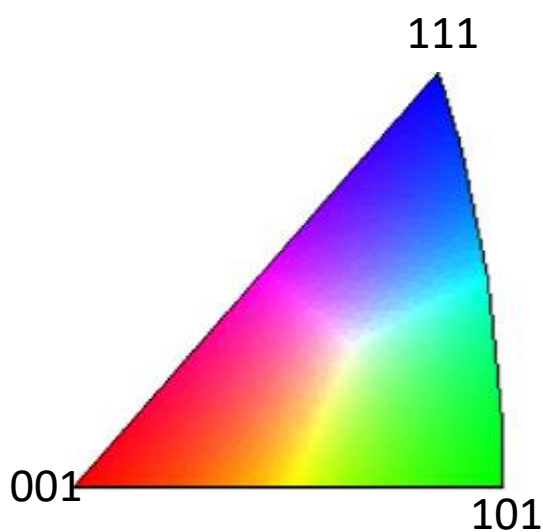


Figure 9: Crystal plane colour indicator

They found out that the microstructure of the laser regions has columnar dendrites that grew epitaxial from the substrate material (Zhao, Lin, Liu, Zhao, Chen, and Huang, 2009) Liu et al also observed the columnar structure of laser glazed regions in their work (Liu, Lin, Huang, Song, Yang, Chen and Huang, 2011) Figure 9 represents crystal plane colour indicator that shows $\langle 001 \rangle$, $\langle 111 \rangle$ and $\langle 101 \rangle$ as possible crystallographic slip planes and directions in both regions. Figures 10, 11 and 12 represent the scanning electron microscopy (SEM) of substrate and laser glazed regions. The microstructure revealed the regions of laser glazed and that of the substrate. In the laser region, there are presences of black spots which can be seen as impurities or inclusions. The presences of bright spots in the substrate are intermetallic substances of heavy metals. Bimodal microstructures were also observed in this region which is interterm as indicated in figure 7 of band contrast imaging.

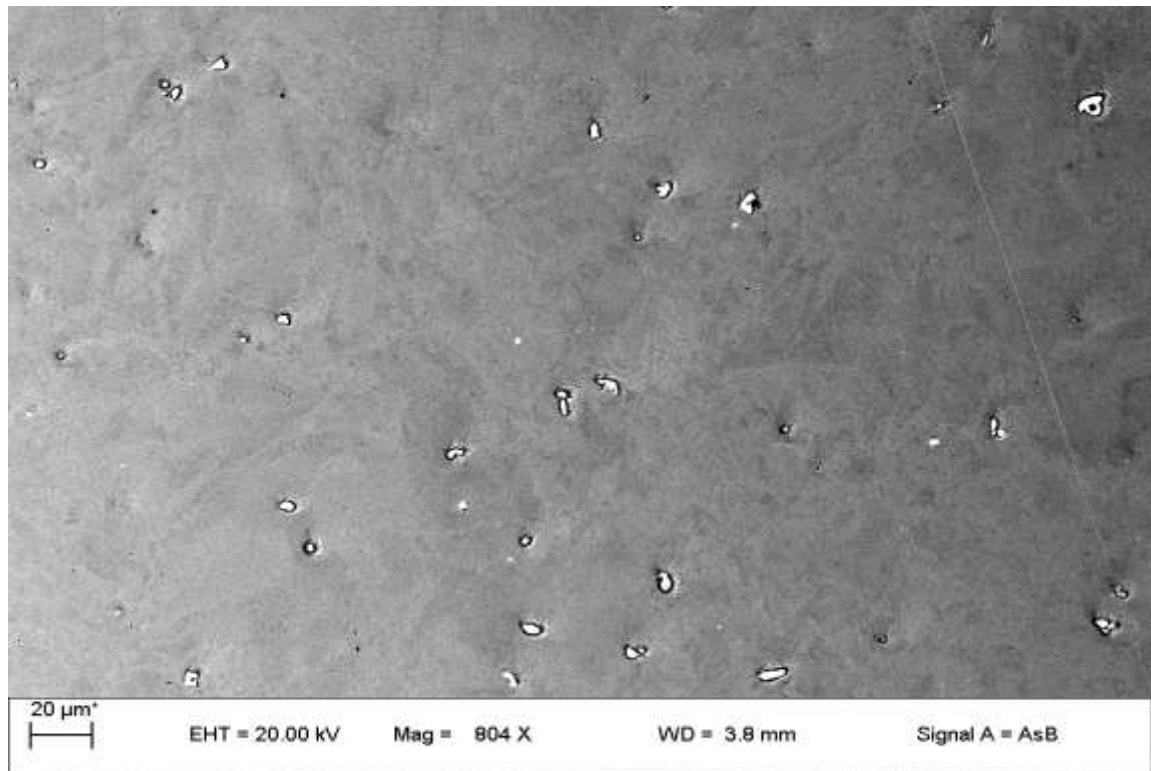


Figure 10 Scanning electron microscopy (SEM) of substrate sample

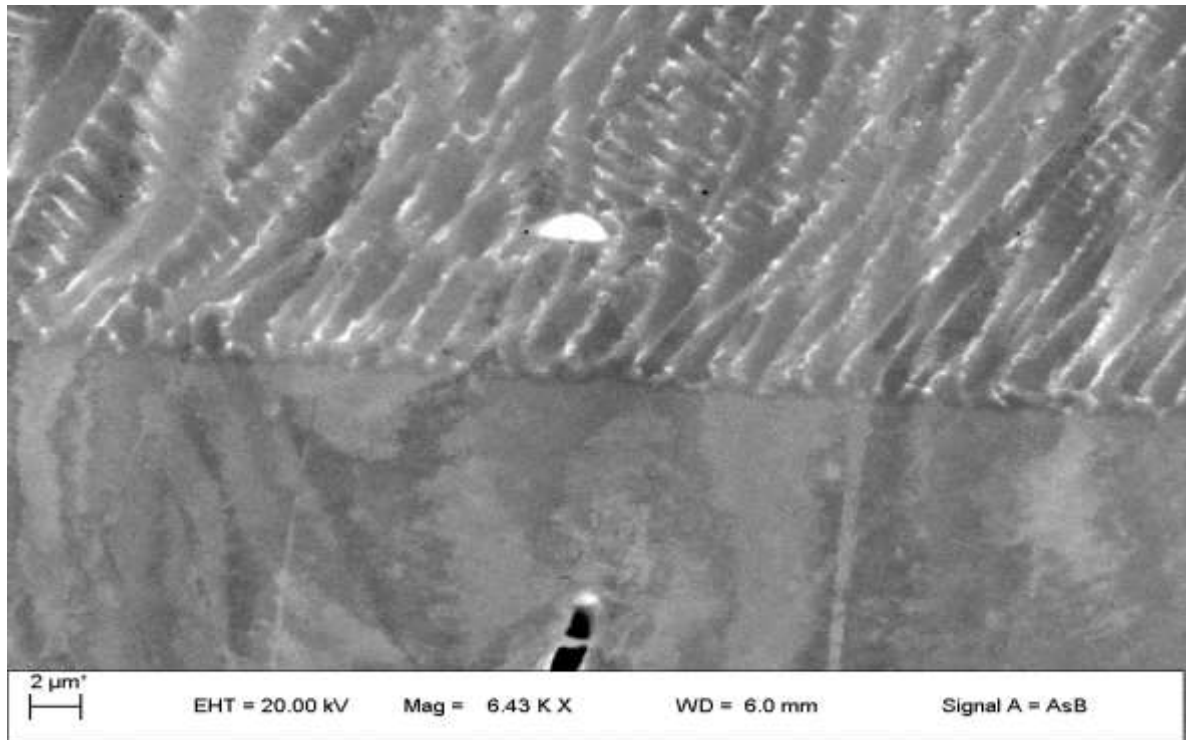


Figure 11 Scanning electron microscopy (SEM) of substrate and laser

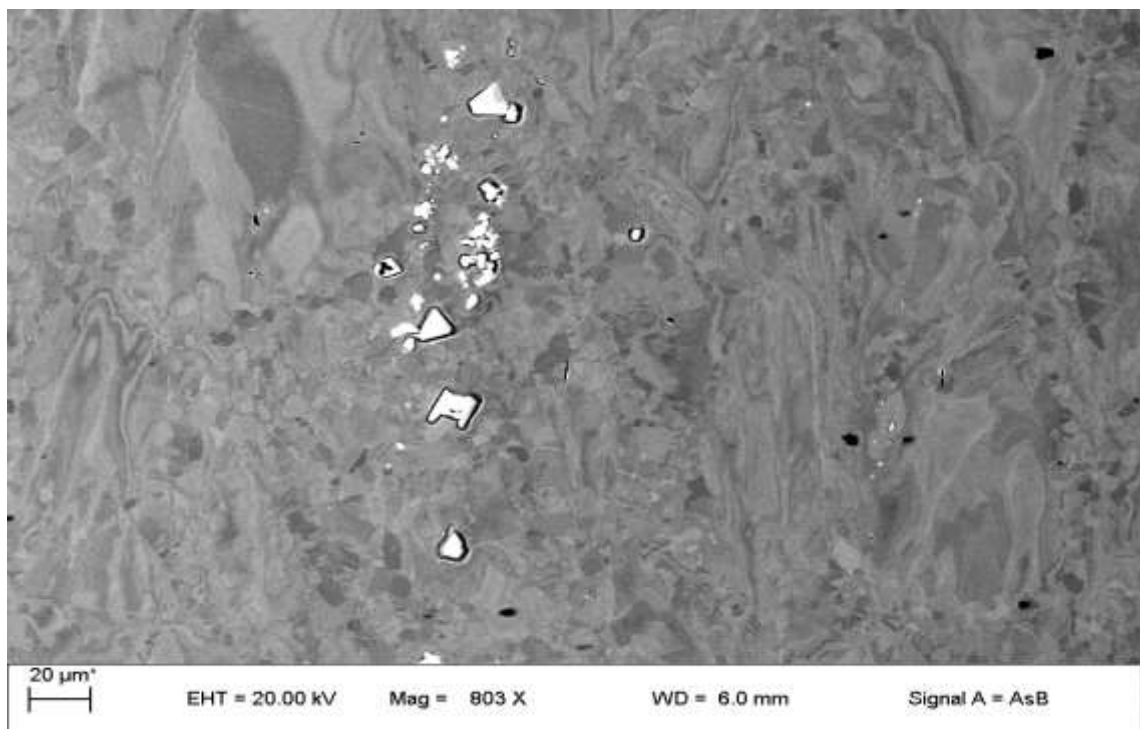


Figure 12: Scanning electron microscopy (SEM) of substrate at higher magnification

The results of energy dispersive x-ray spectroscopy (EDX) of the various elements present in alloy 718 are shown in figure 13. Nickel has the highest weight percent of all the elements present in material. This is in conformity with alloy 718 being referred to nickel base alloy. Other elements present in this material are iron (Fe), chromium (Cr),

niobium (Nb), aluminium (Al), carbon (C), titanium (Ti), molybdenum (Mo). Their weight percent by compositions are as indicated in map sum spectrum of all the elements in the figure below.

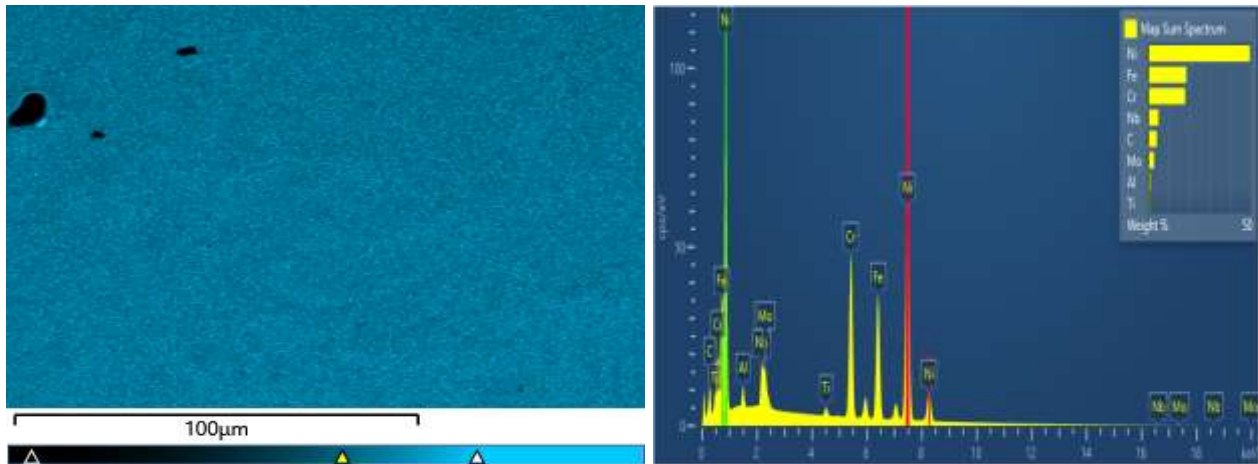


Figure 13: EDX map spectrum of weight percent compositions of elements present in the alloy.

4. Conclusion

Characterizations of laser glazed surfaces and substrate structures of nickel alloy 718 revealed different oxides, microstructural analysis, elemental compositions of the alloy material and possible crystallographic planes and color orientations present in both sections of the material. Conclusively, they show that both surfaces have similar physical and microstructural properties with no interfacial separation at their boundary regions. These indicate that both structures are compatible and should be used for engineering applications.

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