

## The Interface Microstructures and Mechanical Properties of Laser Additive Repaired Inconel 625 Alloy

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**Abstract:** The microstructure and micro-mechanics around the repaired interface, and the tensile properties of laser additive repaired (LARed) Inconel 625 alloy were investigated. The results showed that the microstructure around the repaired interface was divided into three zones: the substrate zone (SZ), the heat-affected zone (HAZ), and the repaired zone (RZ). The microstructure of the SZ had a typical equiaxed crystal structure, displaying simultaneously precipitated block-shaped MC-type carbides (NbC, TiC), with bimodal sizes of approximately 10 nm and 0.5 nm and an irregularly shaped flocculent Laves phase. Recrystallization occurred in the HAZ, and led to significant grain growth; a portion of the second phase dissolved in the original grain boundaries. In the RZ, there was a columnar crystal structure, and the size increased with increasing deposition thickness. Moreover, the microstructure between the layer interface and layer interior was quite different, presenting an overlapping transition zone (OTZ), in which the dendritic structure coarsened and more Laves phase were precipitated, compared to in the layer interior. The hardness and tensile properties of the LARed samples were equivalent to those of the wrought substrate, which indicates that laser additive repairing (LAR) is a reliable repair solution for damaged and mis-machined components comprising Inconel 625 alloy.

**Keywords:** laser additive repairing; Inconel 625 alloy; interface microstructures; micro-mechanics

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### 1. Introduction

Inconel 625 is a nickel–chromium solid-solution strengthened alloy, which is largely strengthened by Nb and Mo [1,2]. It is widely used to fabricate high-temperature components in aeronautical, aerospace, marine, chemical, and nuclear industries, due to its extraordinary combination of high yield and tensile strength, excellent creep strength, and outstanding corrosion resistance at elevated temperature [3–5]. However, these components are easily damaged owing to their aggressive service conditions, and can also be mis-machined during processing [6]. If these damaged and mis-machined components can be repaired rapidly, considerable savings in materials, processing time, and costs can be achieved.

Laser additive manufacturing (LAM) [7] has attracted much attention in the past ten years. By combining laser cladding with rapid prototyping techniques, LAM is an advanced fabrication technique for producing fully dense near-net-shape metallic components on metallic substrates [8]. Based on LAM, laser additive repairing (LAR) technology has been developed [9–11]. By using the damaged or mis-machined components as substrates, the geometry and mechanical properties of the damaged or mis-machined components can be repaired using LAR without deteriorating

the performance of the body parts. Compared with traditional repair technologies [12–14], such as electrobrush plating, thermal spraying, and argon arc welding, LAR has the advantages of a high degree of automation, a small heat affected zone, metallurgical bonding between the repaired zones and degree of automation, a small heat affected zone, metallurgical bonding between the repaired zones the body part, and a reasonable repair cost [15]. A number of metallic components made of different type of materials, including stainless steel, cobalt-based alloys, titanium alloys, and nickel-based alloys, have been repaired using LAR technology [16–18].

In recent years, the processing parameters, microstructures, and mechanical properties of Inconel In recent years, the processing parameters, microstructures, and mechanical properties of Inconel 625 fabricated using LAM technology have attracted much attention [19–22]. Dinda et al. [23] studied Inconel 625 fabricated using LAM technology have attracted much attention [19–22]. Dinda et al. [23] studied the microstructural evolution and structural thermal stability of a LAM Inconel 625 alloy. The results indicated that the as-deposited microstructure mostly consisted of columnar dendrites that were stable up to 1000 °C. Rombouts et al. [24] found that Inconel 625 deposited using directed energy deposition (DED) also showed a microstructure of dendrites parallel to the build direction. Rivera et al. [25] observed fine grain structures at the layer interfaces of Inconel 625 produced using solid-state additive manufacturing. Compared to samples fabricated using LAM, not only the repaired part, but also the microstructures of the interface between the substrate and the repaired part, are essential to the mechanical properties of the component repaired using LAR. The research work of LAR for nickel-based alloys mainly focuses on the repair defects, processing parameters, using LAR. The research work of LAR for nickel-based alloys mainly focuses on the repair defects, microstructures, and mechanical properties of repaired samples. Onuike et al. [10] used DED technology to repair the internal cracks in Inconel 718 alloy. Sui et al. [9] studied the tensile deformation behavior of LARed Inconel 718 alloy with a non-uniform microstructure. To the best of our knowledge, no studies have investigated the relationship between the microstructure and micro-mechanical properties of LARed Inconel 625 alloy around the repaired interface.

In order to explore the potential of LAR as a reliable repair solution for damaged and mis-machined Inconel 625 components, Inconel 625 alloy substrates, with premade trapezoidal shaped defects, were repaired using LAR in this study. The microstructures around the repaired interface and the corresponding mechanical properties were investigated.

## 2. Materials and Methods

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## 2.1. Materials

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The raw materials were prealloyed Inconel 625 powders prepared using a plasma rotating electrode process. These powders had spherical shapes with sizes of 75~125  $\mu\text{m}$ , as shown in Figure 1a.

The microstructure of the powders showed a fine dendritic morphology on the surface, which

1a. The microstructure of the powders showed a fine dendritic morphology on the surface, which was caused by the rapid solidification during atomization (Figure 1b). Before the LAR experiment,

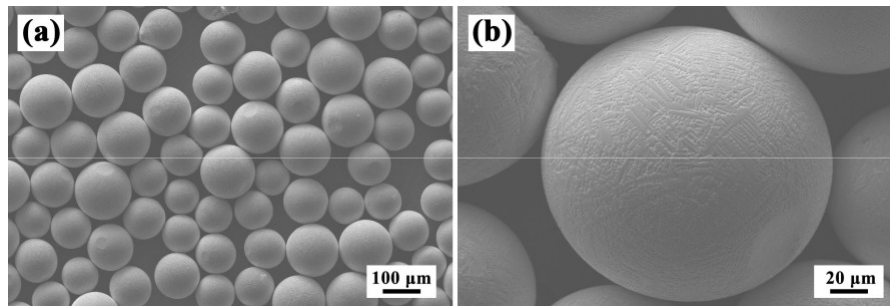
the powders were dried in a vacuum oven at  $120 \pm 10$  °C for 2 h. The LAR process was performed on

the powders were dried in a vacuum oven at  $120 \pm 10$  °C for 2 h. The LAR process was performed on heat treated wrought Inconel 625 substrate, for which the chemical composition is listed in Table 1.

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**Figure 1.** Morphologies of Inconel 625 powders at (a) low and (b) high magnifications.

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**Table 1.** The chemical compositions (wt %) of Inconel 625 alloy powder and substrate.

Material	Ni	Cr	Mo	Nb	Fe	Ti	Al	C	Co	Mn	Si	P
Powder	Bal.	21.92	9.09	3.62	3.71	0.20	0.17	0.036	0.009	0.015	0.06	0.003
Substrate	Bal.	21.70	8.90	3.27	4.40	0.18	0.12	0.030	0.010	0.170	0.15	0.008

Material	Ni	Cr	Mo	Nb	Fe	Ti	Al	C	Co	Mn	Si	P
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2.2. Experimental Procedures

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12000, The LAR experiments were performed on a LAM system (LAM, LSF-Nanjing, China),

Substrate Bal. 21.70 8.90 3.27 4.40 0.18 0.12 0.030 0.010 0.15 0.008

12000, The LAR experiments were performed on a LAM system (LAM, LSF-Nanjing, China), which consists of a fiber laser with a wavelength of 1070 nm, a five-axis numerical control working

table, powder feeder system with a coaxial nozzle, and an inert atmosphere glove box (oxygen content  $\leq 10$  ppm). Argon gas was used to protect the molten pool from oxidation and deliver the alloy The powders LAR experiments were performed on a LAM system (LAM, LSF-12000, Nanjing, China), alloy powders.

which The consists LAR process of a fiber laser performed with a wavelength of 1070 nm, a five-axis numerical control defect, as working shown The LAR process was performed on the substrate with a pre-machined groove defect, as shown

table, in Figure a powder 2a. Then feeder the defect system was with repaired coaxial layer nozzle, b y layer and using an inert the LAM atmosphere system, glove as shown box in (oxygen Figure in Figure 2a. Then the defect was repaired layer by layer using the LAM system, as shown in Figure content 2b. Figure 103 shows ppm).

a Argon schematic gas was of the used scanning to protect strategy the molten of the LAR pool process from oxidation. The scanning and deliver directions the 2b. Figure 3 shows a schematic of the scanning strategy of the LAR process. The scanning directions alloy in the powder s same layer. followed a zigzag pattern, and were rotated by 90 degree in the following layer. in the same layer followed a zigzag pattern, and were rotated by 90 degree in the following layer. The process The LAR parameters process was are performed listed in Table on the 2. substrate with a pre-machined groove defect, as shown in

The process parameters are listed in Table 2.

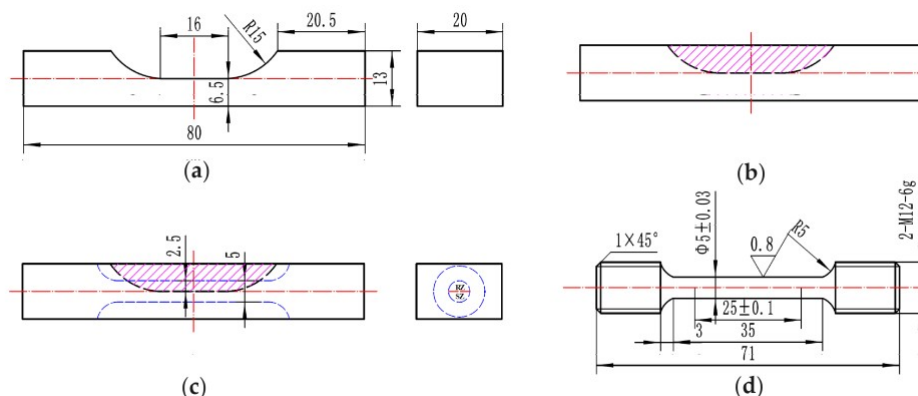
Figure A

2 tensile a. Then testing the defect bar was cut repaired from the layer repaired by layers sample using by the spark LAMerosysion tem., The as shown lume in fraction Figure 2 of b. A tensile testing bar was cut from the repaired sample by spark erosion. The volume fraction of Figure the repair 3 shows zone

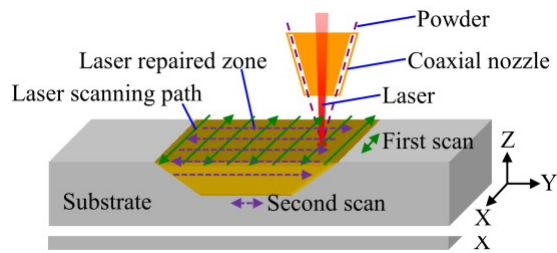
of section the scanning of the tensile stratgy testing of the bar LAR was process 50%, as The shows caning Figure d directions 2c, d. in the

the repair zone in the cross-section of the tensile testing bar was 50%, as shown in Figure 2c, d. same layer followed a zigzag pattern, and were rotated by 90 degree in the following layer. The process

parameters are listed in Table 2.



**Figure 2.** Sketch of (a) substrate with pre-machined groove defect, (b) LARed sample, (c) the position of the tensile testing bar in the LARed sample, and (d) shape and size of the tensile testing bar.



**Figure 3.. Schematic of the scanning strategy of the LAR process..**

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**Table 2.** Process parameters of LARed Inconel 625 alloy.

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Laser Power	Scanning Speed	Laser Spot	Increment of Z	Powder Feeder	Overlaps
Laser Power (kW)	Scanning Speed (mm/min)	Laser Spot Diameter (mm)	Increment of Z Axis (mm)	Powder Feeder Rate (g/min)	Overlaps (%)
1.4	(mm/min) <sub>400</sub>	Diameter <sub>3</sub> (mm)	Increment of Z Axis <sub>0.5</sub> (mm)	Powder Feeder Rate <sub>6</sub> (g/min)	45 (%)
Power (kW)	(mm/min)	Diameter (mm)	Z Axis (mm)	Rate (g/min)	(%)
1.4	400	3	0.5	6	45
1.4	400	3	0.5	6	45

A tensile testing bar was cut from the repaired sample by spark erosion. The volume fraction of

the repaired zone in the cross-section of the repaired bar was examined by optical metallography (OM, Olympus OLS4000, Tokyo, Japan), scanning electron microscopy (SEM, FEI mHetallogrlios Nanolabphy (OM, 600i, Olympus Hillsboro, OLS4000, OR, USA), Tokyo, and Japan), electron backscattered electron microscopy diffraction (EBSD, FEI EDAX, Helios Nanolab 600i, Hillsboro, OR, USA), and electron backscattered diffraction (EBSD, EDAX, Mahwah, NJ, USA). Elemental analysis was carried out using energy-dispersive X-ray spectrometry (EDS, EDAX, Mahwah, NJ, USA). Before the microstructure observation, samples were ground on 500-2000 grit silicon carbide papers and mechanically polished. Polished samples were then electro etched under an 8 V direct current (DC) for 15 s in a reagent of 10% aqueous chromic acid solution to etched under an 8 V direct current (DC) for 15 s in a reagent of 10% aqueous chromic acid solution to Nanolab 600i, Hillsboro, OR, USA), and electron backscattered di raction (EBSD, EDAX, Mahwah, NJ, USA). Elemental analysis was carried out using energy-dispersive X-ray spectrometry (EDS, EDAX, Mahwah, NJ, USA). Before the microstructure observation, samples were ground

on 500–2000 grit silicon carbide papers and mechanically polished. Polished samples were then electro etched under an 8 V direct current (DC) for 15 s in a reagent of 10% aqueous chromic acid solution to reveal the microstructure. All EBSD samples were polished using an argon beam under a voltage of 5 kV and a current of 110 A for 20 min before the EBSD test. The EBSD test used a voltage of 20 kV and a step size of 1  $\mu$ m. X-ray diffraction (XRD, TDF-3000, Dandong, China) scanning was carried out at a constant scanning speed of 10  $^{\circ}$ /min with Cu K radiation, and recorded in 2 $\theta$  ranging from 30 to 100  $^{\circ}$ . To identify tiny phases in the microstructures, a transmission electron microscope (TEM, FEI Titan G2 60-300, Hillsboro, OR, USA) equipped with EDS was applied. The TEM samples, with a 3 mm diameter disc shape, were prepared by mechanical grinding to  $\sim$ 50 nm and then twin-jet polished in a solution of 250 mL HClO<sub>4</sub>+750 mL methanol at a temperature of 30  $^{\circ}$ C and a voltage of 25 V. Vickers hardness of the samples was tested under a load of 200 g and a duration time of 15 s along the deposition direction. The elastic modulus and microhardness of different zones were evaluated by nano-indentation (Hysitron TI-950, Minneapolis, MN, USA) under a load of 5 mN. The room temperature tensile properties of the LARed samples and substrates were tested using a floor model universal testing system (CMT5105, Jinan, China), at a displacement rate of 2 mm/min. For each tensile test condition, three samples were used to calculate the average values and standard deviations of strengths, elongations, and area reductions. The ultimate tensile strength, 0.2% offset yield strength and elongation, was obtained from the resulting stress-strain curves, and the area reduction was determined by measuring the cross-sectional area before and after failure. The fracture morphology was observed using SEM.

### 3. Results and Discussion

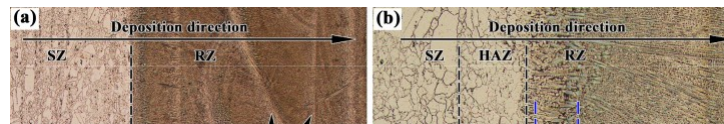
#### 3.1. Microstructure Around the Repaired Interface

##### 3.1.1. Grain Structures

Figure 4a–d show the microstructures in the X–Z section and Y–Z section of the LARed samples around the repaired interface, respectively. The interface between the substrate and the repaired zone presents good metallurgical bonding. No obvious defects, such as cracks and pores can be observed. A transition of microstructure can be observed along the deposition direction, dividing the area into three zones: the substrate zone (SZ), the heat-affected zone (HAZ), and the repaired zone (RZ) (see Figure 4b,d). Inside the RZ, a narrow overlapping transition zone (OTZ) can be observed, between two adjacent deposited layers or tracks (see Figure 4b,d). The width of the OTZ is approximately 0.15 mm. The SZ consists of a mixture of finely and large equiaxed grains. The grains in the HAZ are hard to reveal under the same erosion conditions, suggesting a composition variation in grain boundaries. Detailed grain structure observation was carried out using EBSD, as in the following. RZ presents a columnar dendritic structure that grows epitaxially along the deposition direction. In addition, the dendritic structure in the OTZ between two adjacent deposited tracks is coarser than other areas in the RZ.

Detailed investigations on the microstructures around the repaired interface of the LARed Inconel 625 were carried out using EBSD. Figure 5a–d show the EBSD images in the X–Z section and Y–Z section of the LARed samples around the repaired interface, respectively. The three zones of SZ, HAZ, and RZ can be easily distinguished in the EBSD images, showing grains with different sizes and distributions. The SZ has typical equiaxed grains, with a bimodal grain size distribution. Annealing twins can be observed in the recrystallized grains. Grains in the HAZ grew significantly larger compared to those in the SZ. In the RZ, large sized columnar grains grew epitaxially from the mother grains in the HAZ. The height of the columnar grains can be as large as the layer height. The widths and heights of the columnar grains increase as the deposition height increases. The OTZ cannot be observed in the EBSD maps.

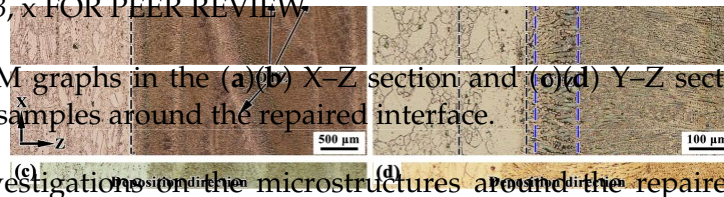
approximately 0.15 mm. The SZ consists of a mixture of finely and large equiaxed grains. The grains in the HAZ are hard to reveal under the same erosion conditions, suggesting a composition variation in grain boundaries. Detailed grain structure observation was carried out using EBSD, as in the following.



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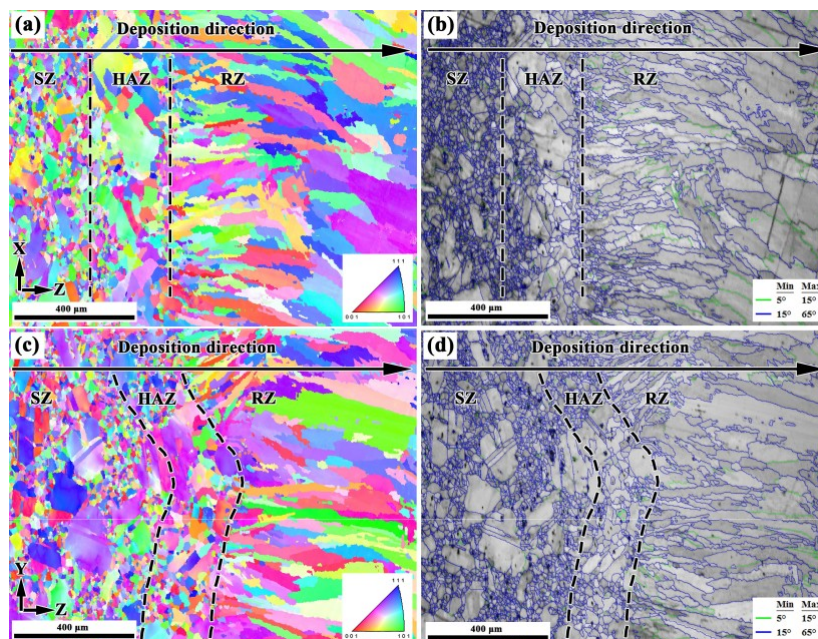
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**Figure 4.** OM graphs in the (a)(b) X–Z section and (c)(d) Y–Z section of LARed Inconel 625 samples around the repaired interface.

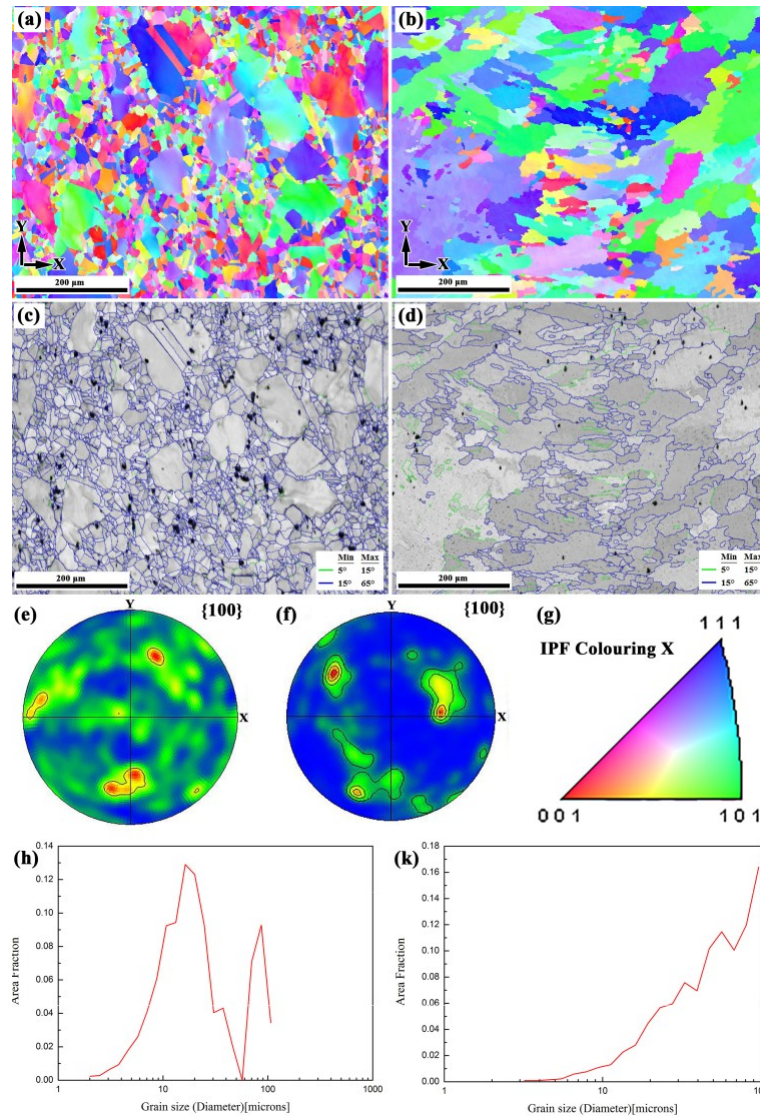


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OTZ



**Figure 5.** EBSD results of the LARed Inconel 625 samples around the repaired interface in the (a)(b)



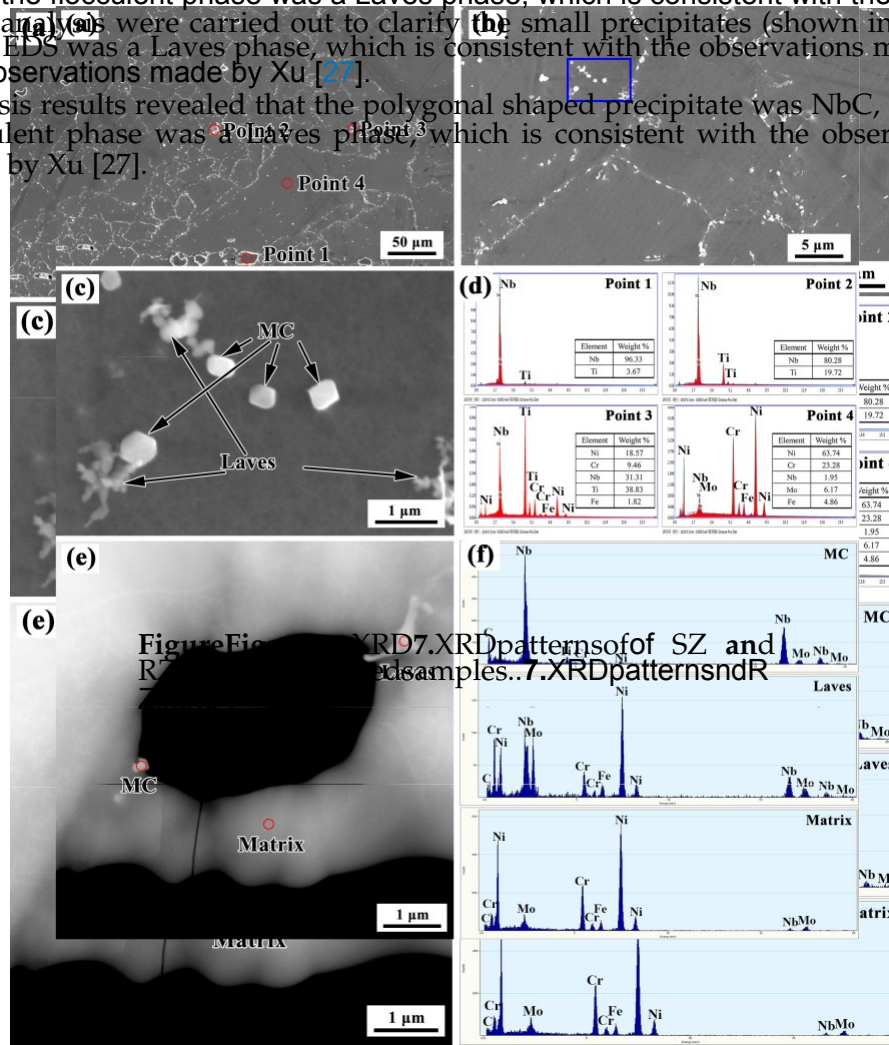
**Figure 6.** EBSD results of LARed Inconel 625 samples of the substrate zone (SZ) (a)(c)(e)(h) and the repaired zone (RZ) (b)(d)(f)(k) in the X–Y section: (a)(b) IPF orientation maps, (c)(d) band contrast maps, (e)(f) {100} pole figures, (g) IPF and (h)(k) grain size distribution.

### 3.1<sup>3</sup>.2<sup>1</sup>. <sup>2</sup>Phases<sup>es</sup>and<sup>and</sup>Precipitations

Figure 7 shows the X-ray diffraction patterns of the RZ and SZ in the repaired sample. It can be demonstrated that the RZ and SZ in the LARed samples both mainly consist of the  $\gamma$  (Ni–Cr) phase

without peaks from other precipitates [26], such as  $\gamma'$  (Ni<sub>3</sub>Al(Ti)),  $\gamma''$  (Ni<sub>3</sub>Nb),  $\delta$  (Ni<sub>3</sub>Nb), Laves (Ni, Fe, Cr)<sub>2</sub>(Nb, Ti, Mo), and carbide phases, due to their low volume fractions or absences. Furthermore,

the diffraction peak intensity of the (200) crystal plane of the  $\gamma$  phase in the RZ is higher than that of the (111) crystal plane, which also indicates that the microstructure in the RZ has a strong texture.



**Figure 8.** (a–c) SEM images, (e) transmission electron microscope (TEM) images, and (d,f) energy-dispersive X-ray spectrometry (EDS) results in the SZ.

**Figure 8. (a–c) SEM images, (e) transmission electron microscope (TEM) images, and (d,f)**

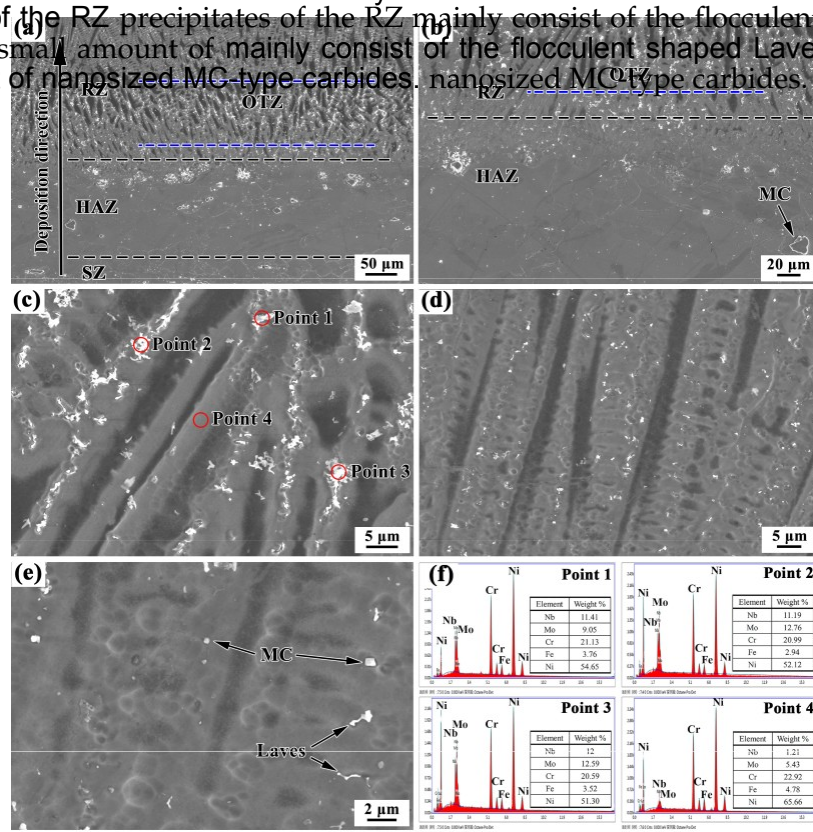
Figure 9a presents the microstructure of repaired samples around the repaired interface. The Figure 9a presents the microstructure of repaired samples around the repaired interface. microstructure is quite different in the SZ and the RZ along the deposition direction. Similarly to the The microstructure is quite different in the SZ and the RZ along the deposition direction. Similarly SZ, a large amount of white precipitates are also seen in the HAZ and RZ. From the previous to the SZ, a large amount of white precipitates are also seen in the HAZ and RZ. From the previous analysis, we can see that the precipitates of the SZ mainly consist of large (approximately 10  $\mu\text{m}$ ) analysis, we can see that the precipitates of the SZ mainly consist of large (approximately 10  $\mu\text{m}$ ) irregular shaped MC-type carbides (M is Nb and Ti), small (approximately 0.5  $\mu\text{m}$ ) polygonal irregular shaped MC-type carbides (M is Nb and Ti), small (approximately 0.5  $\mu\text{m}$ ) polygonal shaped shaped MC-type carbides, and flocculent shaped Laves phase. In the HAZ, there are still many large MC-type carbides, and flocculent shaped Laves phase. In the HAZ, there are still many large irregular irregular shaped MC-type carbides, but some small sized precipitates dissolved in the original grain shaped MC-type carbides, but some small sized precipitates dissolved in the original grain boundaries, as shown in Figure 9b.

as shown in Figure 9b. For the RZ, the microstructure indicates epitaxial growth of the columnar dendritic structure, For the RZ, the microstructure indicates epitaxial growth of the columnar dendritic structure, and a large amount of white precipitates occur along the dendritic boundaries, as shown in Figure and a large amount of white precipitates occur along the dendritic boundaries, as shown in Figure 9a. 9a. In addition, compared to the microstructure in the layer interior, the amount of precipitates is In addition, compared to the microstructure in the layer interior, the amount of precipitates is greater, greater, and the size is largest in the OTZ around the repaired interface, as shown in Figure 9cd. The and the size is largest in the OTZ around the repaired interface, as shown in Figure 9c,d. The precipitates precipitates mainly have flocculent shapes. Figure 9f shows the EDS analysis results of the flocculent mainly have flocculent shapes. Figure 9f shows the EDS analysis results of the flocculent shaped phase shaped phase and the matrix phase. The results of the EDS analysis reveal that the main hardening and the matrix phase. The results of the EDS analysis reveal that the main hardening elements Nb elements Nb and Mo are rich in the flocculent shaped phase, which is the characteristic Laves phase

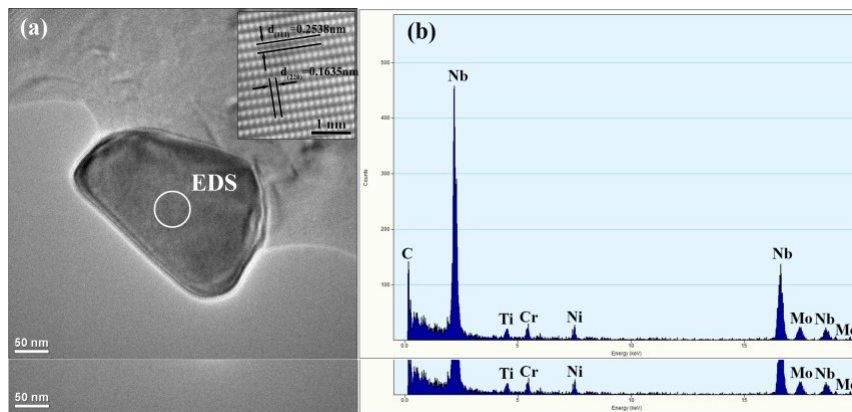
and Mo are rich in the flocculent shaped phase, which is the characteristic Laves phase of Inconel 625 of Inconel 625 alloys [28,29]. This indicates a somewhat heavy segregation in the OTZ. Figure 10 alloys [28,29]. This indicates a somewhat heavy segregation in the OTZ. Figure 10 further shows the further shows the high resolution transmission electron microscopy (HRTEM) image and the EDS

high resolution transmission electron microscopy (HRTEM) image and the EDS results of nanosized results of nanosized precipitates in the RZ. The EDS analysis indicates that it is NbC. Therefore, the

precipitates in the RZ. The EDS analysis indicates that it is NbC. Therefore, the precipitates of the RZ precipitates of the RZ mainly consist of the flocculent shaped Laves phase and a small amount of mainly consist of the flocculent shaped Laves phase and a small amount of nanosized MC-type carbides. nanosized MC-type carbides.



**Figure 9.** Microstructures of LARed samples in different zones: (a) SZ+heat-affected zone (HAZ)+RZ, (b) HAZ+RZ, (c) overlapping transition zone (OTZ), (d, e) RZ, and (f) EDS taken from laves phase and matrix phase of OTZ around the repaired interface.



**Figure 10.** HRTEM images (a) of LARed samples in the RZ, and (b) the EDS results of a NbC precipitate.

Figure 10. HRTEM images (a) of LARed samples in the RZ, and (b) the EDS results of NbC precipitate.

10. HRTEM images (a) of LARed samples in the RZ, and (b) the EDS results of a NbC precipitate.

### 3.1.3. Microstructure in the Overlapping Transition Zone

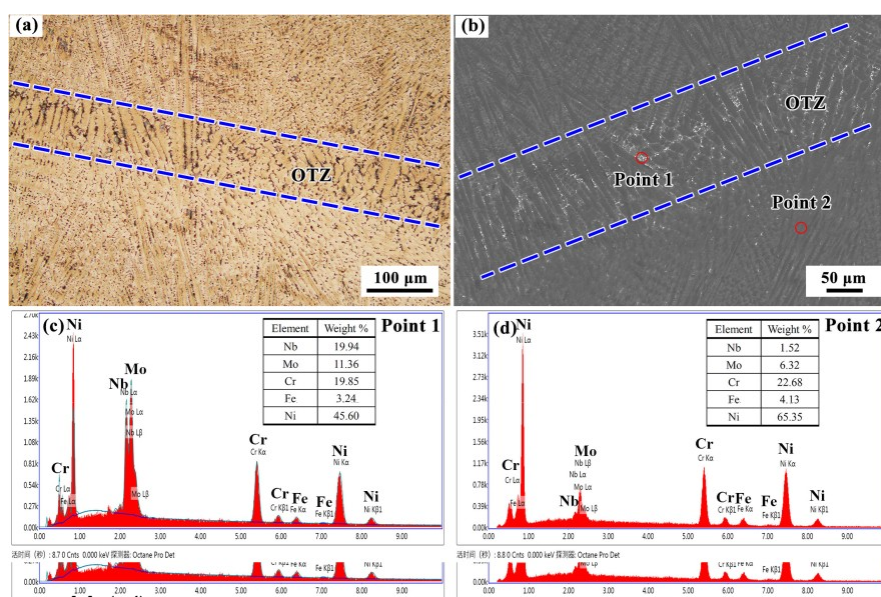
### 3.1.3. Microstructure in the Overlapping Transition Zone

3.1.3. Figure 11 shows the microstructure of the OTZ between two adjacent deposited tracks in the RZ. Similarly to the OTZ around the repaired interface, the microstructure of the OTZ in the RZ also presents a coarse columnar dendritic structure in Figure 11a. The width of the OTZ is also approximately 0.15 RZ. Similarly to the OTZ around the repaired interface, the microstructure of the OTZ in the RZ also approximately 0.15 mm. Particularly, a large amount of white precipitates occur along the dendritic boundaries in the OTZ, as shown in the Figure 11b. The EDS results of the precipitates demonstrate that they are Laves phase, as shown in Figure 11c,d.

OTZ. Similarly to the OTZ around the repaired interface, the microstructure of the OTZ in the RZ also presents a coarse columnar dendritic structure in Figure 11a. The width of the OTZ is also approximately 0.15 RZ. Similarly to the OTZ around the repaired interface, the microstructure of the OTZ in the RZ also approximately 0.15 mm. Particularly, a large amount of white precipitates occur along the dendritic boundaries in the OTZ, as shown in the Figure 11b. The EDS results of the precipitates demonstrate that they are Laves phase, as shown in Figure 11c,d.

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boundaries in the OTZ, as shown in the Figure 11b. The EDS results of the precipitates demonstrate that they are Laves phase, as shown in Figure 11c,d.



**Figure 11.** (a) Microstructure of the OTZ and (b) microstructure of the OTZ and the Laves phase and (c) EDS results of the Laves phase and (d) matrix phases.



according to the Kurz and Fisher analysis [32], the primary dendritic spacing  $\lambda$  can be expressed by a

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function of the dendritic tip radius  $r$  as [33]:

$$\lambda = \frac{\sqrt{3DT}}{rG}, \quad (2)$$

Therefore, the primary dendritic spacing  $\lambda$  is identified as a function of temperature gradient  $G$ . Therefore, the primary dendritic spacing is identified as a function of temperature gradient  $G$  and the growth rate  $R$  as follows:

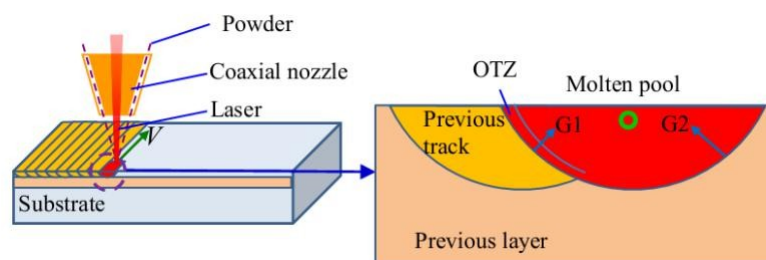
and the growth rate  $R$  as follows:

$$\lambda = \frac{3\pi}{2} \frac{4D_t}{\Delta T} \frac{1}{G} \frac{1}{R} \quad (3)$$

$$= \frac{6\pi D_t}{kR} \quad (3)$$

The product  $G \cdot R$  (cooling speed) governs the size of the solidification structure [34].

Thus, the The product  $G \cdot R$  (cooling speed) governs the size of the solidification structure [34]. Thus, difference in the temperature gradient  $G$  and the growth rate  $R$  during the LAR process should be the difference in the temperature gradient  $G$  and the growth rate  $R$  during the LAR process should be the primary reason for the different morphologies in the OTZ. During the LAR process, the material the primary reason for the different morphologies in the OTZ. During the LAR process, the material is deposited track-by-track and layer-by-layer. When a new track is deposited, the top portion of the is deposited track-by-track and layer-by-layer. When a new track is deposited, the top portion of material in the previous track is remelted, and then forms a molten pool, as shown in Figure 12. The the material in the previous track is remelted, and then forms a molten pool, as shown in Figure 12. The temperature of the previous track is higher than that of the previous layer, therefore, the temperature The temperature of the previous track is higher than that of the previous layer, therefore, the temperature gradient at the overlapping line,  $G_1$ , is less than at the other fusion line,  $G_2$ . Consequently, the  $G_2R$  Consequently, the  $G \cdot R$  (cooling rate) is lower at the overlapping line and higher at the other fusion line. This leads to a line. This leads to a significant increase in the primary dendritic spacing  $\lambda$  along the overlapping line significant increase in the primary dendritic spacing along the overlapping line of the molten pool, of the molten pool, which forms an OTZ between tracks. which forms an OTZ between tracks.



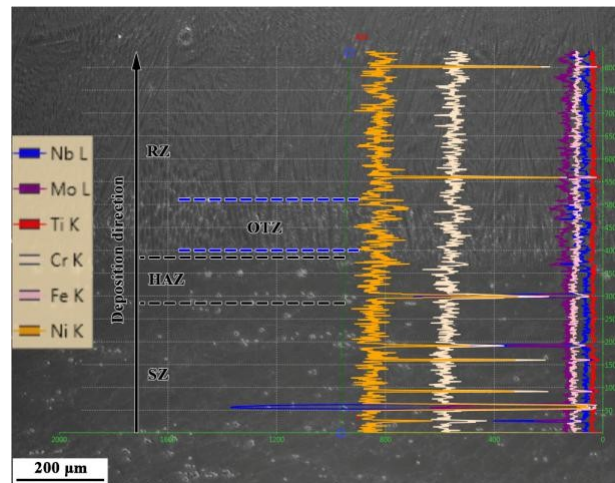
**Figure 12.** Schematic illustration of the temperature gradient,  $G$ , and growth rate,  $R$ , distribution in the molten pool during the LAR process.

The size of the Laves phase is attributed to the concentration of the alloying elements. During the LAR process, solidification in the Inconel 625 alloy starts with the primary liquid  $\rightarrow \gamma$  reaction, causing the **accumulation** of Nb, Mo, C, and Ti in the interdendritic and grain boundary liquids. Thus, the Laves phase and MC and -type MC carbides can precipitate in these regions. Then, the subsequent liquid  $\rightarrow \gamma + \text{NbC}$  reaction consumes most of the available carbon until another eutectic reaction  $\gamma + \text{Laves}$  occurs, finishing Laves occurs, the solidification process [27]. The Laves process phase is an unavoidable terminal phase in solidification of Inconel 625 alloys. However, in Inconel 625 alloys, conditions of solidification strongly influence the extent of niobium segregation and the amount of Laves phase [35]. Thus, of a slow cooling rate, (GR) in a slow OTZ cooling causes a large  $(G \cdot R)$  in segregation, the OTZ causes the alloying large segregation elements, of which all lead to the formation of a large amount of Laves phase.

### 3.2.2. Composition Distribution

The EDS line scanning results show that the major alloying elements, such as Ni and Cr, are distributed uniformly from the SZ to the RZ, as shown in Figure 13. The fluctuations of some elements are mainly due to the detection of precipitations. However, the Nb and Mo elements are distributed more uniformly in the RZ compared to the SZ. This is mainly due to the segregation of Nb and Mo elements in the SZ to form a large amount of MC-type carbide and Laves phases, while the degree of segregation of the alloying elements in the RZ is weak, and the precipitated phase is slow. In addition, the degree of segregation of the alloying elements is aggravated in the OTZ, compared to the layer interior, this is due to Nb and Mo elements segregating and forming a large amount of Laves phase.

compared to in the layer interior, this is due to Nb and Mo elements segregating and forming a large amount of Laves phase.



**Figure 13.** EDS line scanning results of the LARed Inconel 625 alloy sample along the deposition direction.

**Figure 13.** EDS line scanning results of the LARed Inconel 625 alloy sample along the deposition direction.

### 3.3. Mechanical Properties

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##### 3.3.1. Hardness

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Figure 14 shows the Vickers hardness along the deposition direction, around the repaired interface.

The hardness values show have the

no Vickers notable hardness difference along between the end of the deposition the SZ and direction, RZ. The average

und average the hardness repaired is approximately 240 HV values.

have no notable difference between the SZ and RZ. The average

The micro is approximately 240 properties  $\pm 20$  HV of the LARed Inconel 625 alloy samples were evaluated using a nanoindentation.

mechanical for the reduced properties modulus of the (ELARed) and microhardness Inconel 625 alloy

around the repaired interface using. The nanoindentation for load the-

depth reduced curves modulus of the matrix ( $E$ ) and phase microhardness different zones ( $H$ )

of around the LARed the repaired samples

interface are shown. The nanoindentation 15. The responses load-depth of

the curves LARed of the Inconel matrix 625 phase alloy are in predominantly different zones plastic, of the LARed although samples some elastic are

shown recovery in upon Figure release 15a. The load responses can also of

be observed LARed. Inconel both figures, 625 alloys small are

plateaued dominantly can be seen plastic, at the although maximum some load, elastic which recovery is indicative upon release of creep of in the the load Inconel can also 625 be alloy observed.. In both The figures, resultant small load-depth plateau curves can be seen used at the to maximum the H load, and which  $E_r$  by employing is indicative the of procedure creep in the of Inconel Oliver and 625 Pharr alloy. [36]. The  $E_r$  was then used, together with Poisson's ratio ( $\nu$ ), to calculate the elastic modulus The resultant ( $E$ ) from the load following-depth curves equation: were used to obtain the  $H$  and  $E_r$  by employing the procedure of Oliver and Pharr [36]. The  $E_r$  was then used, together with Poisson's ratio ( $\nu$ ), to calculate the

elastic modulus ( $E$ ) from the following equation

$$\frac{1}{E_r} = \frac{1}{E} + \frac{1 - \nu_i^2}{E_i}, \quad (4)$$

where  $E_i$  is the elastic modulus of the indenter (1140 GPa) [37], its Poisson's ratio (0.07) and  $\nu_i$  is the Poisson's ratio used for the Inconel 625 alloy was set to 0.303 [38]. The obtained

wh indentation  $E_r$  is the modulus elastic (modulus  $E_r$ ) and microhardness of the indenter ( $H$  (1140 data GPa) of the and LA Red  $\nu_i$  is Inconel 625 ratio alloy (0 sample.07)[37] in. Furth different more, zones the are Poisson's shown in Figure ratio used 15b. for The the  $E_r$  values Inconel for 625 di alloy erent was zones set reveal to 0.303 imperceptible [38]. The obt changes in.

The indentation microhardness modulus of (the  $E_r$ ) matrix and microhardness phase in the RZ ( $H$ ) is data approximately of the LA Red 5.1

GPa, Inconel which 625 is alloy slightly sample higher in different than that of zones the SZ are (approximately shown in Figure 4.8 GPa) 15b.. This  $E_r$  is

due values to the for weakening different zones of the segregation reveal imperceptible of the Nb changes and Mo. alloying The microhardness elements in the of RZ the.

In addition, trix phase the in microhardness the RZ is approximately of the matrix 5 phase. 1 GPa, in the which HAZ is slightly generally higher demonstrates than that

low the value SZ (approximately 44.8.6 GPa).. This is attributed due to the to weakening the dissolution of the of segregation the MC-

type of carbide the Nb and and Laves Mo alloying phases and elements the growth in the of RZ the. In grains addition, in the the HAZ microhardness. In particular, of the

microhardness six phase in of the HAZ matrix generally phase in

demonstrates the OTZ is approximately low value 4.7

(approximately GPa, which is slightly 4.6 GPa) lower. This than is attributed that of layer to in the interior dissolution in the RZ of (approximately the MC-

typical carbide 5.1 GPa) and. The Laves formation phases

and of abundant the growth Laves of the phases grains in the OTZ HAZ is due. In

to particular, the large these segregation microhardness of alloying of the elements

Nb phase and in Mo. The OTZ Laves is approximately phase is brittle 4.7

GPa, harmful which phase, is and slightly consumes low

than large that amount of layer of Nb interior and Mo, in and the decreases RZ (approximately the a

mount 5 of 1 Nb GPa) and. The Mo

formation in the matrix, of abundant which leads Laves to the phases decrease in the of OTZ the micro

hardness is due to the large of these segregation matrix phase of alloying the OTZ elements.

Nb and Mo. The Laves phase is a brittle harmful phase, and consumes a large amount of Nb and Mo,

and Materials decreases the amount of Nb and Mo in the matrix, which leads to the decrease of the microhardness of the matrix phase in the OTZ. and decreases the amount of Nb and Mo in the matrix, which leads to the decrease of the microhardness of the matrix phase in the OTZ.

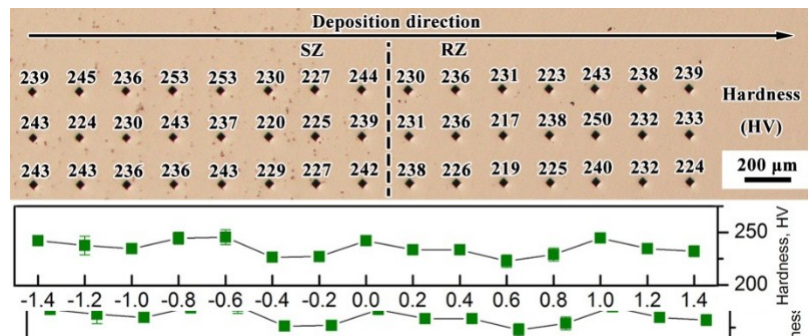


Figure 14. Vickers hardness values of the LARed Inconel 625 alloy sample.

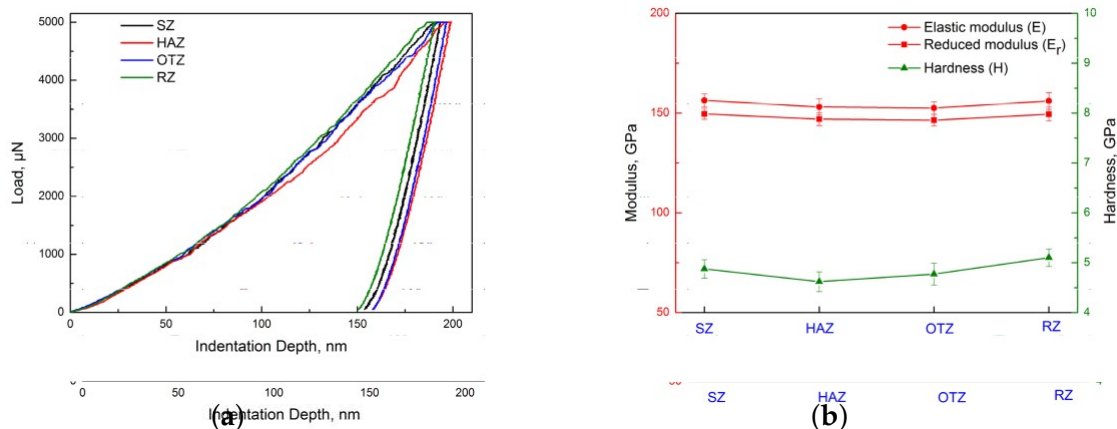


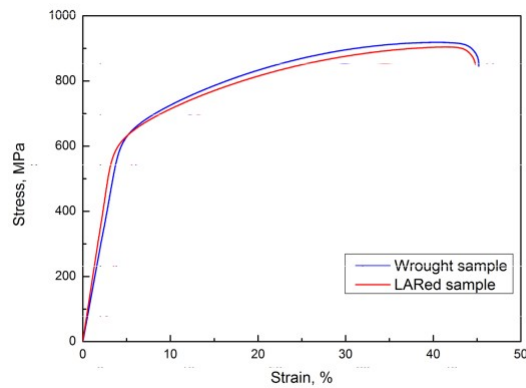
Figure 15. (a) Nanoindentation load-depth curves and (b) indentation modulus and hardness data of the LARed Inconel 625 alloy sample.

Figure 15. (a) Nanoindentation load-depth curves and (b) indentation modulus and hardness data of the LARed Inconel 625 alloy sample. The room temperature tensile properties of the LARed sample and the heat-treated wrought samples are listed in Table 3. The tensile strength and ductility of LARed samples are similar to those of the wrought substrate, which indicates that LAR is a reliable approach for damaged and mis-machined components made of the Inconel 625 alloy. To compare the tensile behaviors of the samples in the various conditions, tensile testing of

Table 3. Room temperature tensile properties of wrought and LARed Inconel 625 alloy samples.

mis-machined components made of the Inconel 625 alloy. Tensile testing of the LARed and wrought samples was done, and the results are shown in Figure 16. The tensile curves for the different samples are almost the same, which indicates that they have similar tensile properties. The LARed sample shows a slightly steeper elastic response than the wrought sample.

Number stress-strain plastic sample curves 908 elongation ±5	Strength in the period before fracture. 44.67 ± 0.5	5870.2(Mpa)±3	Reduction 45.0 ± 0.7	
	σ <sub>b</sub> (Mpa)		Ψ (%)	
Wrought substrate	914±4	572±6	45.3 ± 0.4	43.7 ± 0.3
LA Red sample	908±5	587±3	44.7 ± 0.5	45.0 ± 0.7
Wrought substrate	914±4	572±6	45.3 ± 0.4	43.7 ± 0.3



**Figure 16.** Typical tensile test curves of LARed sample and wrought substrate.

**Figure 16.** Typical tensile test curves of LARed sample and wrought substrate

Figure 17 shows the fractographs of the wrought sample. The fracture surface of the wrought

Figure 17 shows the fractographs of the wrought sample. The fracture surface of the wrought sample exhibits fine dimples, which indicate a ductile mode of failure associated with good tensile

sample exhibits fine dimples, which indicate a ductile mode of failure associated with good tensile properties. In addition, a large number of secondary cracks can be found. The fractograph of the

properties. In addition, a large number of secondary cracks can be found. The fractograph of the wrought sample shows a distinct morphology, with MC-type carbides inside the larger dimples. At the wrought sample shows a distinct morphology, with MC-type carbides inside the larger dimples. At

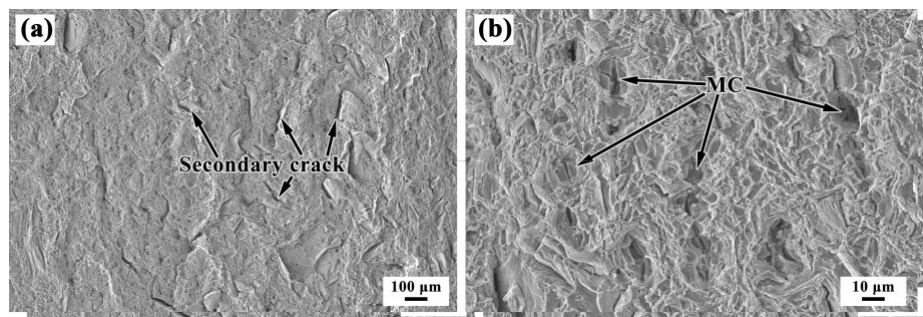
wrought sample shows a distinct morphology, with MC-type carbides inside the larger dimples. At same time, the second phase MC-type carbides are torn, as shown in Figure 17b. It is evident that the same time, the second phase MC-type carbides are torn, as shown in Figure 17b. It is evident that

the same time, the second phase MC-type carbides are torn, as shown in Figure 17b. It is evident that the MC-type carbides are primarily responsible for making the fracture process easier by providing

the MC-type carbides are primarily responsible for making the fracture process easier by providing favorable sites for excessive microvoid initiation, and the growth of macroscopic cracks.

favorable sites for excessive microvoid initiation, and the growth of macroscopic cracks.

favorable sites for excessive microvoid initiation, and the growth of macroscopic cracks.



**Figure 17.** Fractographs of the wrought substrate sample at (a) low and (b) high magnifications.

**Figure 17** Fractographs of the wrought substrate sample at (a) low and (b) high magnifications.

Figure 18 shows the fractographs of the LARed sample. A mixed fracture occurred in the samples, which included the SZ and RZ, and the fractographs are quite different, as shown in Figure 18a. Figure 18b shows the high-magnification SEM image of the SZ, which is similar to the wrought sample, where the fracture of the SZ contains a dimpled surface indicative of ductile failure, and a surface dimpled indicative of ductile failure. The dimples and tear edges in the RZ fracture have a clear orientation, as shown in Figure 18c,d. Since the direction of the tensile stress at this time is almost perpendicular to the direction of the dendrite growth in the sample, the aligned dimples and tear edges also indicate that the microstructure of the LARed samples has a distinct orientation in the deposition direction. When the sample is stretched perpendicularly to the deposition direction, the dendritic structure is cut transversely, leaving a dimpled structure, with the dendrite center and dendrite dry areas as the torn edge. As the dendrite growth and dendrite column arrangement is regular, a rupture is left after the regular arrangement of the dimples.

regular, regular, arrangement, rupture is left after the regular arrangement of the dimples.

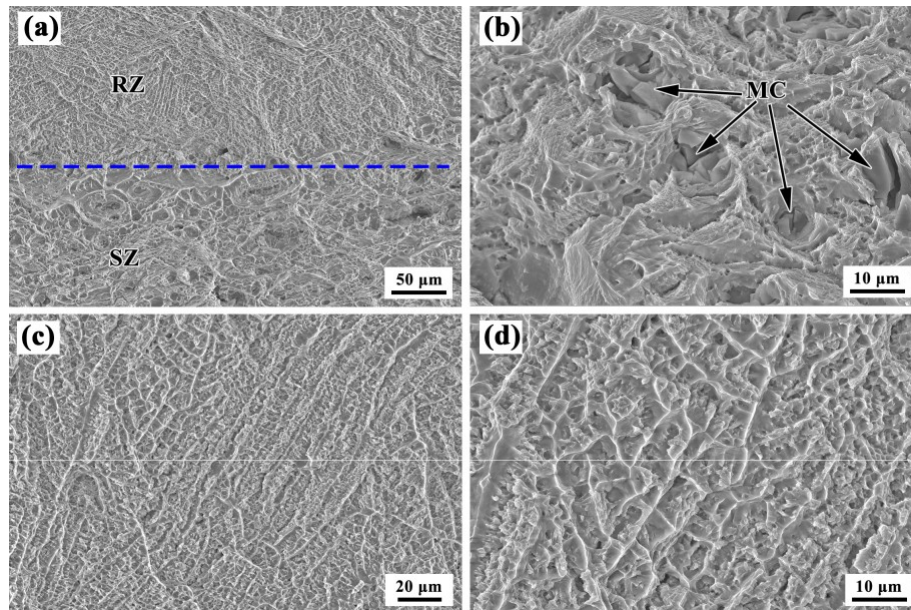


Figure 18. Fractograph of the LARed sample: (a) SZ+RZ, (b) SZ, and (c,d) RZ.

Figure 18. Fractograph of the LARed sample: (a) SZ+RZ, (b) SZ, and (c,d) RZ.

#### 4. Conclusions

In this paper, Inconel 625 alloy substrate with premade trapezoidal groove shaped defects were repaired using LAR. The microstructures str t s around the repaired interface and the corresponding mechanical properties were investigated. The conclusions are outlined below:

- The microstructure around the repaired interface of LARed Inconel 625 alloy can be divided into three zones: the SZ, HAZ, and RZ. The SZ has typical equiaxed crystal structure with bimodal grain distribution, while in the HAZ, recrystallization occurs and leads to significant grain growth. In the RZ, there are very large columnar grains, and the size of the columns increases with the number of deposited layers.
- The precipitates in the SZ mainly consist of large (approximately 10 μm) and small (approximately 0.5 μm) block-shaped MC (MnS) type Nb carbides and (Mn, Ti) shaped irregularly flocculent Laves phase. In the HAZ, there are still many large MC-type block carbides, but some precipitates dissolve in the original grain boundaries. For the RZ, some Laves phase and a few MC-type carbides are precipitated.

type along carbides the epitaxial are precipitated growth dendritic along boundary the epitaxial  
es. growth dendritic

The boundary microstructure i.e. between two adjacent deposited tracks presented an overlapping transition

- The microstructure (OTZ), which is between dendritic two structure adjacent coarsened, deposited and tracks more represented Laves phases overlapping precipitated compared transition to zone in the (OTZ), layer which interior the. The dendritic of structure the OTZ was coarsened, approximately and more 0.15 Laves mm. The phase lower precipitated the OTZ compared led to an increase the layer in interior dendrite. The arm width spacing of the and OTZ the form was approximately of the Laves 0.15 phase mm. The lower The G-R in Vickers the OTZ hardness led to an increase indentation dendrite modulus arm spacing from the and SZ the to formation the RZ along of the the Laves deposition phase.

- The direction Vickers hardness were not notably different indentation the modulus LARed samples, from the SZ were to the approximately RZ along the 240 deposition 20 HV. The direction microhardness were not notably of the matrix different phase in the LARed RZ (approximately samples, and were 5.1 GPa) approximately was slightly higher  $\pm 20$  than HV. that The of microhardness the SZ (approximately of the matrix 4.8 GPa), phase while in the RZ microhardness approximately generally 5.1 GPa) achieved was slightly lower value higher (approximately than that of the 4.6 GPa) (approximately in the HAZ. In particular, GPa), while the the microhardness of the generally matrix achieved phase in the lower OTZ value was (approximately 4.7 GPa, GPa) which in the was HAZ slightly. In lower particular, than that the of microhardness layer interior of in the RZ matrix (approximately phase in the 5.1 OTZ GPa) was. The approximately fluctuation of the 4.7 microhardness GPa, which was of the slightly matrix lower phase than around that of the repaired layer interior interface the was RZ mainly (approximately caused by the 5.1 segregation GPa). The fluctuation of the Nb and Mo microhardness alloying elements of the.

matrix phase around the repaired interface was mainly caused by the segregation of the Nb and

- The yield strength and elastic modulus of the LARed samples were higher than those of the wrought sample. The tensile strength and ductility of the LARed samples were similar to those of the wrought sample. Both the SZ and RZ presented a dimple fracture surface, and a large number of secondary cracks could be found in the SZ. The dimples and tear edges had a clear orientation of the wrought sample. Both the SZ and RZ presented a dimple fracture surface, and a large number of secondary cracks could be found in the SZ. The dimples and tear edges had a clear orientation in the RZ. The comprehensive tensile properties of the LARed Inconel 625 alloy are equivalent to those of the wrought alloy.

**Author Contributions:** Y.W., Q.X. and G.L. conceived and designed the experiments; Y.W. and L.Y. performed the experiments; Y.W., Q.X. and G.L. analyzed the data; R.L. and W.W. contributed analysis tools; Y.W. wrote the paper. All authors have read and agreed to the published version of the manuscript.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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