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HEAT TREATMENT EFFECT ON PHYSICAL PROPERTIES OF STAINLESS STEEL / INCONEL BONDED BY DIRECTED ENERGY DEPOSITION

In this study, stainless steel 316L and Inconel 625 alloy powders were additively manufactured by using directed energy deposition process. And heat treatment effect on hardness and microstructures of the bonded stainless steel 316L/Inconel 625 sample was investigated. The microstructures shows there are no secondary phases and big inclusions near interfacial region between stainless steel 316L and Inconel 625 except several small cracks. The results of TEM and Vickers Hardness show the interfacial area have a few tens of micrometers in thickness. Interestingly, as the heat treatment temperature increases, the cracks in the stainless steel region does not change in morphology while both hardness values of stainless steel 316L and Inconel 625 decrease. These results can be used for designing pipes and valves with surface treatment of Inconel material based on stainless steel 316L material using the directed energy deposition.

Keywords: Directed Energy Deposition, Interface, Physical properties, Heat treatment

1. Introduction

Additive Manufacturing (AM), also known as 3D printing, is a promising technology that manufactures parts by stacking organic/inorganic materials using digital design data [1-3]. Additive manufacturing processes using metal materials such as powder are classified into Powder Bed Fusion (PBF) and Directed Energy Deposition (DED) processes. The PBF process is advantageous for producing precise and complex parts, and the DED process has advantages in terms of large-sized products, repair, and maintenance compared to the PBF process. In particular, in the case of parts that simultaneously shape or join two or more materials, the DED method is the only additive manufacturing process [4,5].

The DED process is mainly applied to the surface treatment as well as the building of the entire parts such as surface-hardening or maintenance [6-8]. An expensive nickel-based alloy such as Inconel has a drawback of inferior economic efficiency to use after processing it into a single material. Therefore, the practical applications of Inconel are trend using for the surface exposed to the internal/external environment and which replacing the rest region of part with inexpensive materials. For example, for accessories such as pipes and valves in contact with

seawater, the material of the outer diameter portion responsible for rigidity and the inner diameter portion requiring chemical and abrasion resistance must be different. The conventional technology was difficult to manufacture parts by using different materials together, whereas the introduction of the DED process can overcome these difficulties.

Despite the interest in the bonding of different materials through the DED process [9,10], the studies of the process parameters or microstructure such as cracks of build structures have been limited compared to the PBF process. Although the DED-joined bulk structures with nickel-based materials which are known to have good bonding properties with iron-based materials have wide applications, the research of DED process have only conducted for the controlling process parameters such as laser power or gas supply rate [9-12]. In particular, the research field of the bonding interface formed by constructing iron-based powder (316L stainless steel alloy, STS 316L) and nickel-based powder (IN 625) has been little studied.

Thus, in this study, both interfacial microstructures including cracks and Vickers hardness near the interface were investigated to the bulk tubular-structure manufactured by the DED process using two materials of STS 316L and IN 625.

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2. Experimental

The DED process (Directed Energy Deposition, InssTek Co., South Korea) was performed using IN 625 powder (53-150 μm) and STS 316L powder (45-150 μm) provided by KOSWIRE Co. (Korea). The STS 316L is a low-carbon steel and has excellent intergranular corrosion resistance in a welded state [13]. The IN 625 that have resistance to oxidation and corrosive environments, is mainly used in marine equipment such as piping and valves for chemical and pollution prevention facilities [14]. The process parameters of DED additive manufacturing are unique data secured by InssTek Corporation. The STS 316L material was used as the substrate, and a pipe-shaped body (Fig. 1(a)) of IN 625 inside and STS 316L outside was additively manufactured.

The fabricated bulk structure prepared by mounting and polishing process for the microstructure analysis was observed using the field emission scanning electron microscope (FE-SEM, JSM-7100F, JEOL Co., Japan) and the scanning transmission electron microscope (STEM, JEM-ARM200F, JEOL Co., Japan). The Fe and Ni elements distribution of the surface was performed by the EDS (Energy Dispersive X-ray Spectroscopy) mapping. Vickers hardness (HM-211, Mitutoyo Co., Japan) was measured to confirm the change of hardness around the interface onto the polished specimen. The load of indenter tip was set to 3.92 N and the holding time was set to 10 seconds. In order to the physi-

cal properties change due to heat treatment, the bulk structures were heated up to 1065, 1130, and 1200°C at the heating rate of 10°C/min. The specimens were holding for 90 min at each set temperature and the finally water-cooled.

3. Results and discussion

Fig. 1(a) shows a photograph of a bulk structure in which dissimilar materials of IN 625 (inside) and STS 316L (outside) are additively manufactured in a pipe form onto a substrate made of STS 316L. Fig. 1(b) shows the cross-sectional SEM image at the interface between the both materials in the fabricated pipe. It was found that the interface appears to be sound and there are no detachment between two materials. However, there were several cracks with a few tens of micrometer in the STS 316L region in the samples as shown in Fig. 1(c), which must be defects caused by the difference in coefficient of thermal expansion (CTE) of the two materials. That is, since the difference in CTE values causes tensile stress in the STS 316L region during laser melting and solidification of DED process, the cracks can be formed [15].

Fig. 1(d) shows that there are fine pores of about 200 and 400 nm inside STS 316L and IN625 region, respectively. It is analyzed that these spherical nano-pores are normally originated from the trapped pores contained in the initial metal alloy powder,

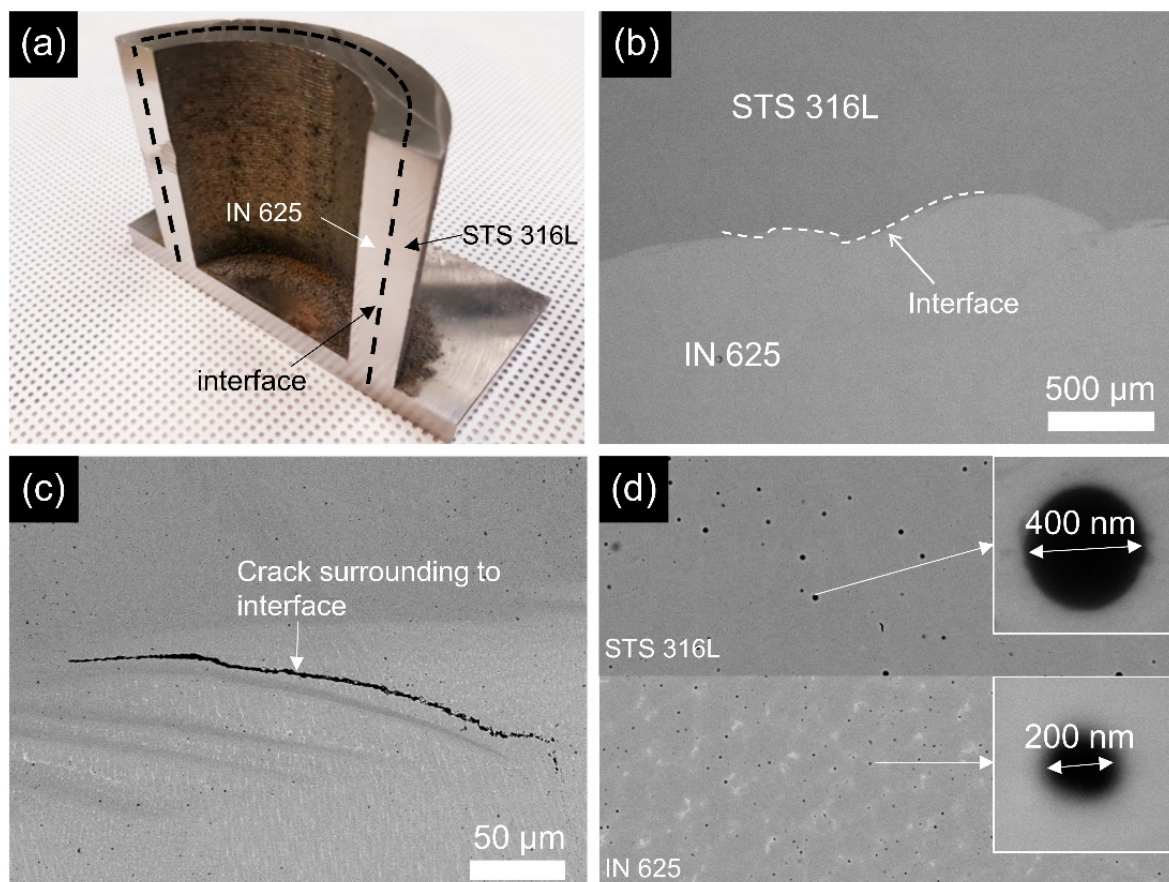


Fig. 1. (a) A photo of dissimilar metal pipe of IN 625 and STS 316L materials manufactured by DED process, (b) microstructure image and (c) observed crack of IN 625/STS 316L interface area, (d) a image of macro-pore inside the both materials

or that gas that could not escape during the laser melting process was captured as it was after cooling. The observed pore size of STS 316L is relatively large compared to that of IN 625 in the whole sample. The origin of this different pore size could be described due to that the thermal conductivity of each material is also different. It is known that the high thermal conductivity of stainless steel compared to that of Inconel 625 provides rapid cooling rate [9] that does not contribute further diffusion of voids near pores. Thus, it is analyzed that the pore size has become relatively large in STS 316L.

Fig. 2 shows TEM results on interfacial region between STS316L/IN625 materials. As shown in Fig. 2(a), there were no visible defects, and they were well-bonded like one material. The area indicated by the dotted line is estimated by the interface, and it was possible to clearly distinguish the interface through EDS analysis as shown in Fig. 2(b). Fig. 2(c) and (d) shows high magnification TEM images on STS 316L and IN625 region and there were no big inclusions or defects in both materials. It is also analyzed that the crystal plane of two materials is corresponding to [111]. And the lattice distance of 0.208 nm in Ni-based materials was observed as shown in Fig. 2(d).

Fig. 3(a) shows comparison of cross-sectional SEM images with heat-treatment. The effect on the interface due to heat treatment does not appear to be significant. Interestingly, it was still found that several large cracks were not removed with a heat

treatment as shown in Fig. 3(b). Considering the temperatures of 1065 and 1200°C are fit for STS 316L and IN 625, respectively, we also introduced intermediate temperature of 1130°C as heat-treatment for interface. Fig. 3(c) shows comparison of Vickers hardness values with heat-treatment. Heat treatment was performed at 1065, 1130, and 1200°C, respectively, and water-cooling was immediately performed thereafter. In order to measure the hardness without being affected by each other, the indenter tip was taken with a sufficient distance at intervals of 0.7 mm. As shown in Fig. 3(c), it was confirmed that the hardness value changed greatly around the bonding interface. When the thickness of the mixed zone is inferred approximately as hundreds μm through the change of the hardness value. All heat-treated samples show lower Vickers hardness value than as-built sample. When heat-treatment temperature is 1065°C, average hardness value of STS 316L decreases from 223 Hv to 186 Hv. And it is measured that the value at 1130°C is somewhat less reduced. This tendency is similarly observed in IN 625 region. However, both materials show big drop in hardness values at 1200°C heat treatment. Hence, if there are no problem in the interfacial region, it can be described that the heat treatment temperature should be considered between 1130 and 1200°C. This phenomenon appears to be the difference in microstructure growth according to the heat treatment temperature, and the size of the grains was measured.

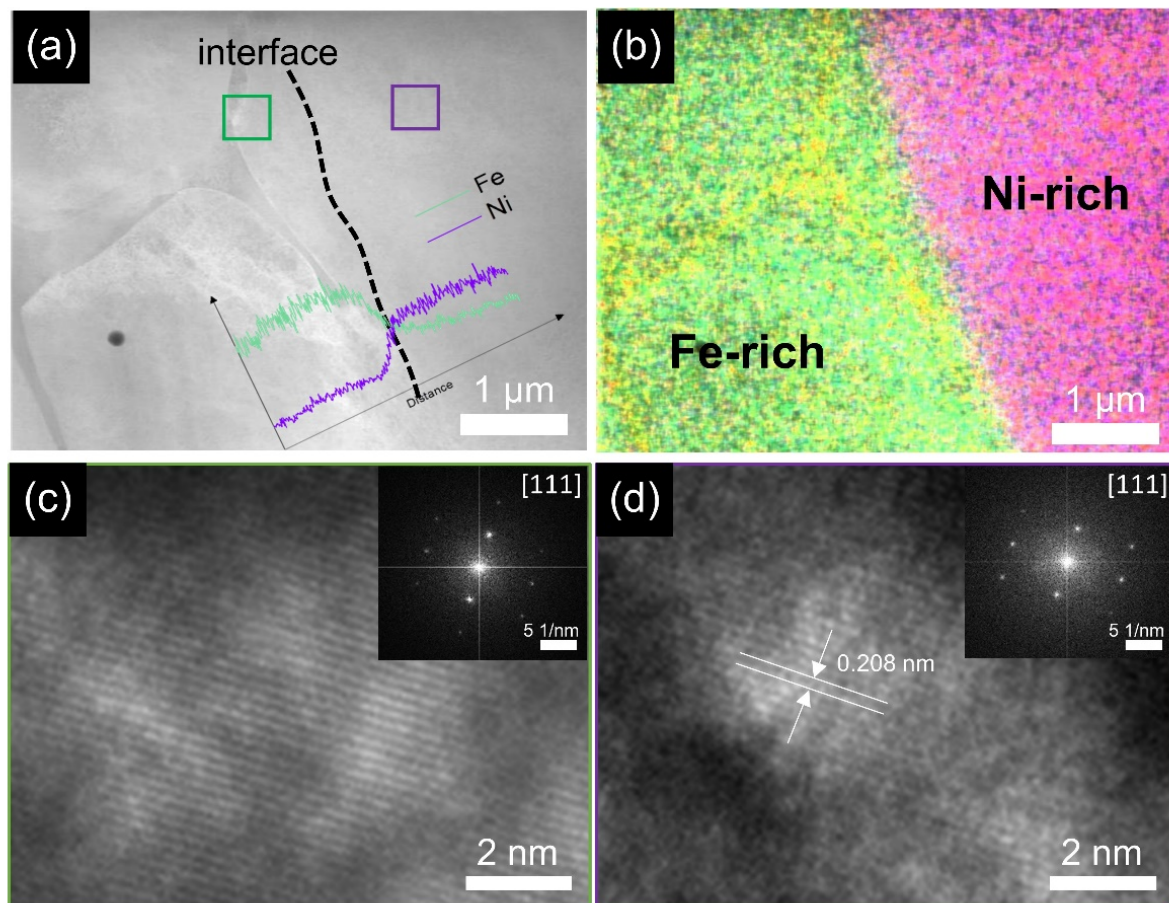


Fig. 2. (a) A cross-sectional TEM image of the IN 625/STS 316L interface area and line EDS (inset) analysis result, (b) EDS mapping distribution of the Fe and Ni elements in the interface area, and HR-TEM images and SAED patterns of the (c) Fe-rich and (d) Ni-rich zones from the both sides of the interface

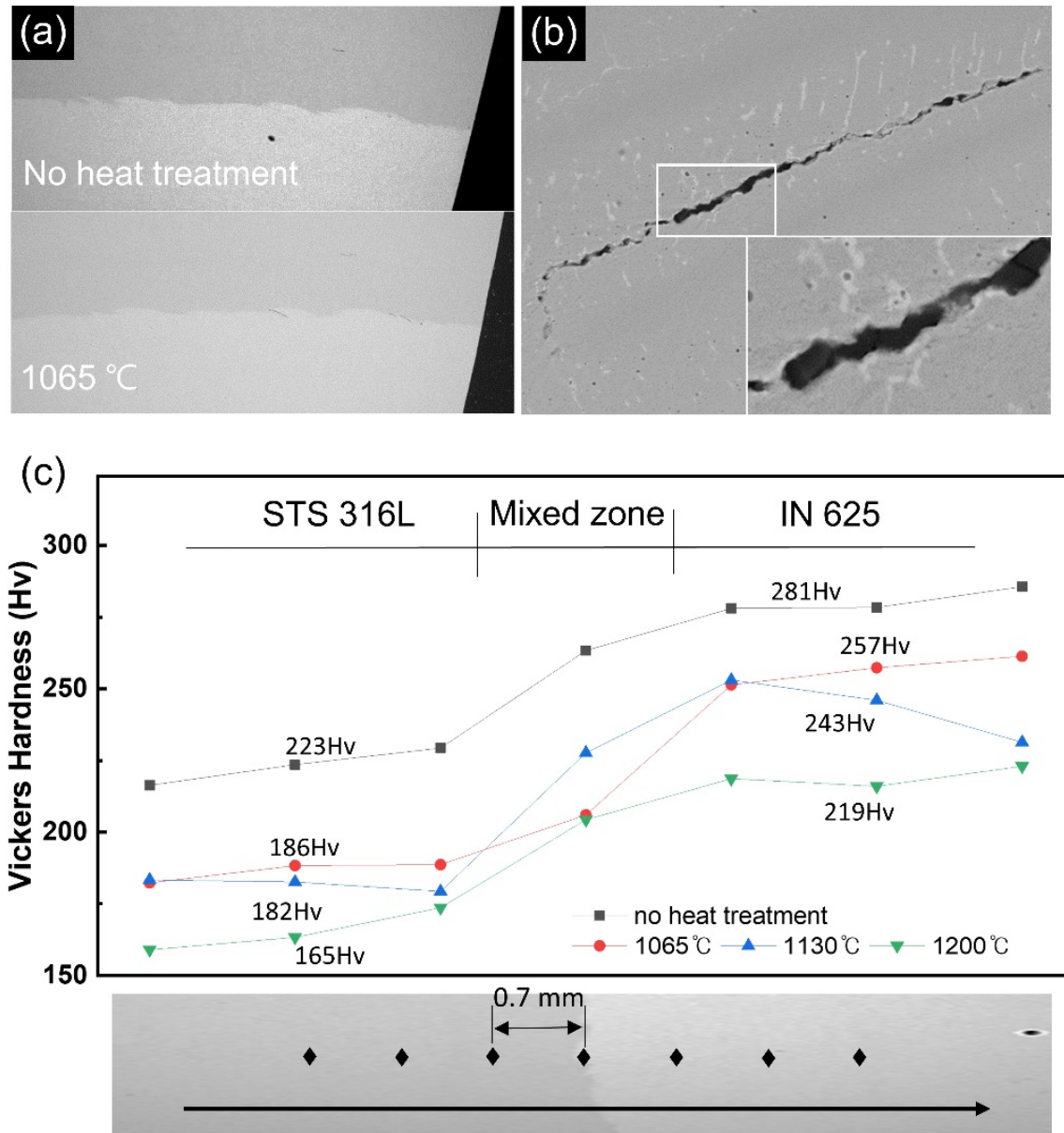


Fig. 3. FE-SEM images of the interface area of (a) the dissimilar metal pipe with/without heat treatment and (b) including crack after heat treatment, and (c) Vickers hardness changes of the dissimilar metal pipe as heat treatment and the position of practical measured indenter tip

Fig. 4(a)~(d) show microstructures in which the characteristics of grains inside STS 316L are displayed. There was a clear difference between microstructures according to the heat treatment. In Fig. 4(e), the average grain size measured by EBSD equipment is shown according to the heat treatment temperature. As the heat treatment temperature increased, the average grain size increased from 42 to 85 μm . This change in the average grain size can explain the results in Fig. 3(c). The Hall-petch equation (Eq. (1)), which is a general correlation between grain boundaries and hardness, is as follows.

$$H_v = H_0 + K_H d^{-1/2} \quad (1)$$

Here, H_v is the hardness value, H_0 and K_H are constants depending on the hardness, and d is the size of the grain. It is possible to explain the decrease in hardness value due to heat treatment through Eq. (1). It can be seen that as the heat treatment tempera-

ture increases, grain growth occurs, the average size increases, and thus the hardness value decreases. As shown in Fig. 4(e), the grain sizes of STS 316L become similar due to grain growth caused by high heat-treatment temperature at 1130 and 1200°C, respectively. Therefore, even though grain growth of STS 316L occurs, the temperatures of 1065 and 1130°C should be considered as appropriate post-heat treatment condition in viewpoint of less drop in hardness of IN 625.

4. Conclusions

In this study, the effect of heat treatment on the change of microstructures and harness in the bonding interface was confirmed in samples obtained by additively manufacturing stainless steel alloy powder (STS 316L) and Inconel alloy

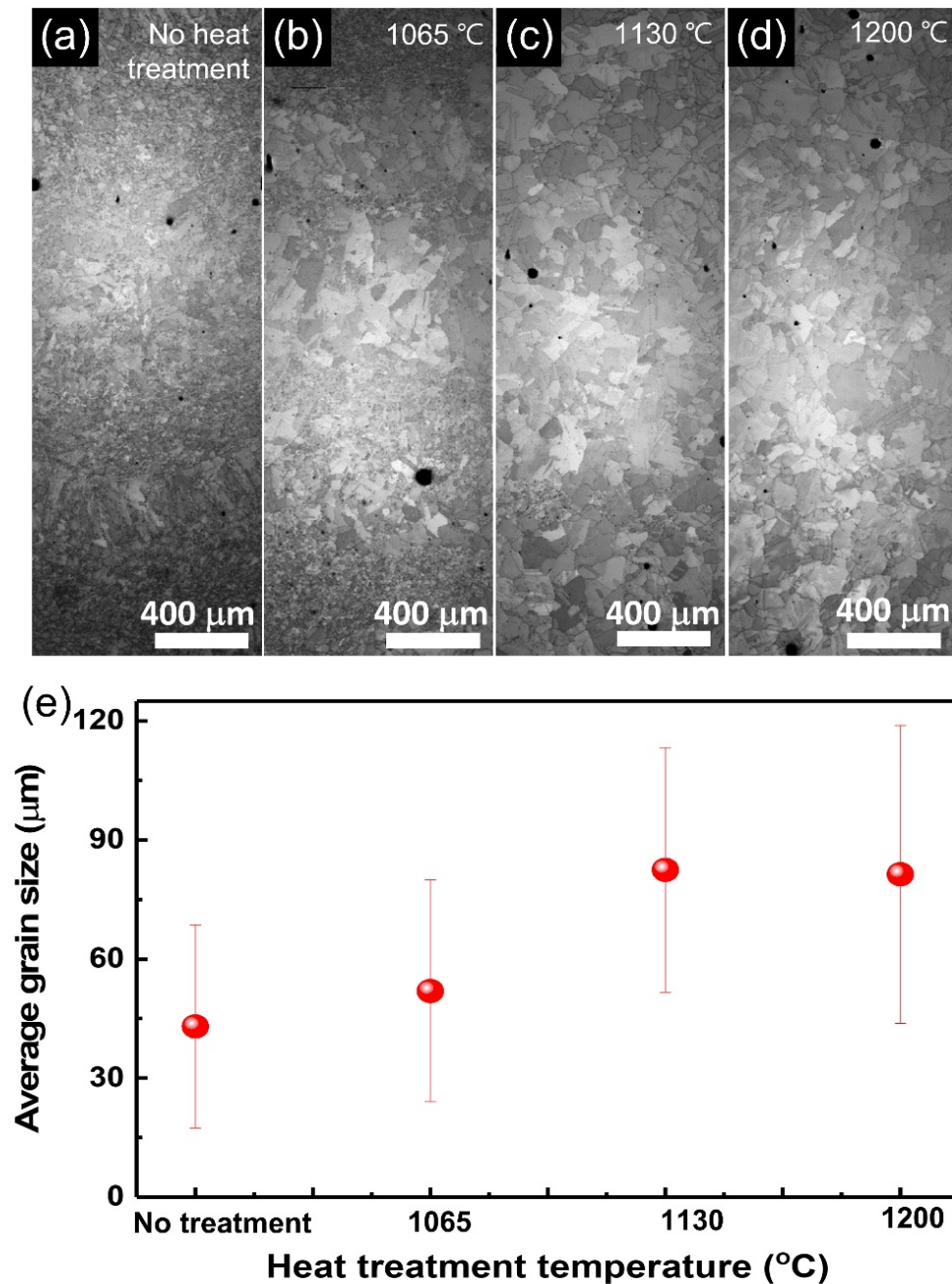


Fig. 4. Grain microstructure images of (a) without heat treatment, after heat treatment at the (b) 1065°C, (c) 1130°C, (d) 1200°C and (e) average grain size changes of the dissimilar metal pipe according to heat treatment temperature

powder (IN 625) by DED process. Overall, it can be seen that the Fe-based stainless alloy and the Ni-based Inconel alloy are directly bonded, and even though the interface bonding area is very narrow, no abnormal structure at the interface was found. Furthermore, post-heat-treatment experiment was clearly performed to check for achieving sound interfacial microstructures as well as suitable mechanical performance. Hardness values in both STS 316L and IN 625 are sensitively changed, which appears to be a difference in grain growth according to the heat treatment temperature. These results can be used as fundamental information for designing pipes and valves with surface treatment of Inconel material based on STS 316L material using the directed energy deposition process.

Acknowledgments

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