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ORIGINAL ARTICLES

OPTIMIZATION OF A METALLOGRAPHIC METHODOLOGY FOR THE STUDY OF LASER-MELTED LAYERS OF AUSTENITIC STAINLESS STEEL

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ABSTRACT

INTRODUCTION: After laser melting during welding and heat treatment processes, the melted zone of the austenitic steel is characterized by a specific fine-grained microstructure formed as a result of the high heating and cooling rates. This causes additional difficulties when the microstructures of the molten zone and the base metal have to be investigated.

AIM: The aim of the present paper is to optimize the methodology for preparation of metallographic samples in order to study the microstructure of a laser-melted layer and of a base metal on the same sample.

MATERIALS AND METHODS: Samples of AISI 321 austenitic stainless steel with laser-melted surface layers were ground and polished by a conventional procedure, but etched with aqua regia for different time periods.

RESULTS AND DISCUSSION: It is found that the microstructure of the laser-melted layers is revealed after a shorter exposure time (2 min) of the reagent, as longer exposure time leads to over-etching. A longer exposure time (5 min) is required to reveal the microstructure of the base metal. Subsequent polishing of the sample results in more obscure structural features.

CONCLUSION: An optimized procedure of a two-stage etching process is proposed. In the first stage, the microstructure of the laser-melted layer is revealed in 2 min exposure time, followed by metallographic studies. After that the second stage continues with additional etching of 3 min to reveal and study the microstructure of the base metal.

Keywords: *metallographic methodology, austenitic stainless steel, laser-melted layer, microstructure*

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INTRODUCTION

Austenitic stainless steels are one of the first materials for production of implants and medical devices (1,2). They are characterized by relatively good mechanical properties and high corrosion resistance. These steels contain between 12 and 30% chromium as alloying element. Their corrosion resistance is due to the single-phase austenite structure and the passivating effect of chromium (3). Due to the pres-

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ence of the chromium in the content, a thin Cr_2O_3 layer is formed on the surface of the steel, which protects the structure from the development of corrosion processes.

Austenitic stainless steels are suitable for dental purposes due to their high corrosion resistance and non-magnetic properties. They are subeutectoid alloys of Fe with C and the alloying elements Cr, Ni, Ti, Mo, and Ta (3). The good mechanical properties and high corrosion resistance are defined by alloying with various elements. Carbon leads to hardness increase, but to corrosion resistance decrease, hence in the austenitic steels it should be in a minimum amount (3). With carbon content higher than 0.030%, carbides of the $Cr_{23}C_6$ type are precipitated along the grain boundaries, which leads to a decrease in the Cr amount in the adjacent areas (1). As a result, the corrosion resistance decreases, especially at the grain boundaries. Chromium is a ferrite stabilizer, while nickel is an austenite stabilizer. In the presence of nickel, chromium must be a minimum of 18% for an austenitic structure to exist at room temperature (3). Steels of the AISI 302B, 316, 317, 321, and 347 type, which contain Si, Mo, Ti, and Nb, have δ-ferrite in their structure due to the ferrite stabilizing effect of these elements (4).

Depending on the type of treatment, different phases may exist in the structure of the austenitic stainless steels: ε-martensite and α′-martensite upon cooling to very low temperatures or intense plastic deformation; different types of carbides ($M_{23}C_6$, M_6C) depending on the alloying elements and the type of heat treatment; Laves phases during prolonged holding at high temperatures; σ-phase during cold plastic

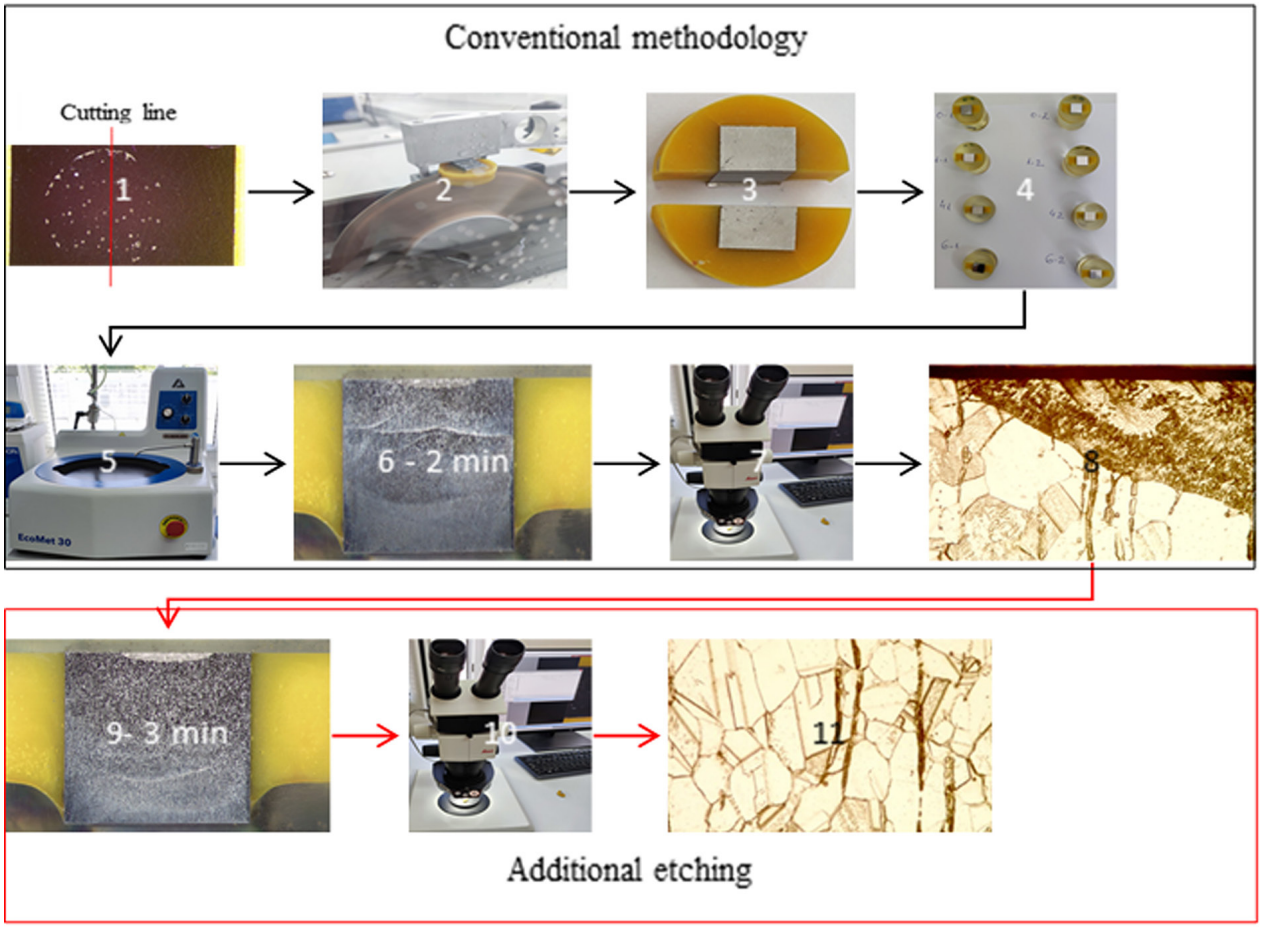


Fig. 1. Flowchart of metallographic methodology, consisting of conventional methodology and additional etching. Conventional methodology stages: 1) sample observation; 2) sample cutting; 3) cut sample; 4) specimens in epoxy resin; 5) grinding and polishing; 6) sample etching; 7) OM observation; 8) microstructure of melted layer. Additional etching: 9) additional etching of base metal; 10) OM observation; 11) microstructure of base metal.

deformation (4). All these structural elements react differently to the etching reagents. Therefore, a universal etching solution for revealing all these phases could not be found. Different reagents are used depending on the type of steel and the structural element to be investigated (4,5). In some cases, successive etching processes take place.

After laser melting during welding and heat treatment processes, the melted zone of the austenitic steel is characterized by a specific fine-grained microstructure formed as a result of the high heating and cooling rates (6). This causes additional difficulties when the microstructures of the molten zone and the base metal have to be investigated.

AIM

The aim of this paper is to optimize the methodology for the preparation of metallographic samples in order to study the microstructure of a laser-melted layer and of a base metal on the same sample.

MATERIALS AND METHODS

Metallographic samples of AISI 321 (EN X6CrNiTi 18-10) austenitic stainless steel were made in order to study the microstructure of laser-melted layers on their surface. The steel chemical composition was as follows: 0.075% C; 18.20% Cr; 10.85% Ni; 0.98 Si; 1.82% Mn; 0.042% P; 0.012% S; 0.52% Ti, and the rest (weight %)—Fe.

The initial samples were prismatic with dimensions of 10x30x100 mm^3. One surface of each specimen was melted with a continuous CO_2 laser with different modes of laser treatment shown in our previous study (7). Samples with dimensions of 10x10x10 mm^3 were cut from them and subjected to electrochemical corrosion in Ringer solution with parameters described in the work of Dikova Ts. et al. (7).

To study the microstructure and the cracks development in depth of the laser-melted layers, it is necessary to prepare cross-sectional samples. For this purpose, the samples were cut along the diagonal of the area subjected to corrosion tests, or in the maximum proximity to corrosion defects (Fig. 1). Cutting was performed on a Buehler IsoMet 15HC/IsoMet 1000 precision saw cutting machine with a Buehler IsoCut wafering blade high concentration diamond disc (Table 1). The as-received samples were poured with epoxy resin in special molds and were prepared for further processing.

Each of them was grinded and polished with a Buehler EcoMet 30 machine. The grinding was done successively with P 400, P 800, P 1200, and P 1500 sandpapers. After grinding with the given sandpaper, the sample was washed abundantly with running water and dried with hot air. Polishing was performed in 3 stages according to the manufacturer's instructions (Table 1). First, the specimens were polished with an UltraPad disc and MetaDi Supreme diamond suspension (9 μm), then with a TriDent disc and MetaDi Supreme diamond suspension (3 μm), and finally with a ChemoMet disc and MasterPrep aluminum final polishing suspension. After each

Table 1. Processes and consumables for preparation of metallographic samples.

№	Process	Consumable	Note/Abrasive	Velocity, [rpm]	Time, [min]
1.	Cutting	Diamond disk	Counterweight 250 g	375	~25
2.	Grinding	Sandpaper (P400) Sandpaper (P800) Sandpaper (P1200) Sandpaper (P1500)	To flettness	300	
3.	Polishing	Stage 1 UltraPad	Diamond suspension MetaDi Supreme, 9 μm	150	5
		Stage 2 TriDent	Diamond suspension MetaDi Supreme, 3 μm	150	3
		Stage 3 ChemoMet	Suspension for final polishing MasterPrep Alumina	150	2

polishing step, the samples were washed successively with water and ethanol and air-dried.

The polished samples were etched with aqua regia ($3HCl + 1HNO_3$) (5). The revealing of the microstructure was controlled on a Leica M 80 optical microscope at different magnifications (7.5; 10; 20; 40; and 60) and during different time intervals (30 s, 1 min, 2 min, 3 min, 4 min, and 5 minutes). The microstructure study was done with Leica M 80 optical microscope with Leica IC 90 E digital camera, Olympus SZ51 and XJL-17A optical microscopes.

RESULTS

The conventional methodology for preparation of metallographic specimens of austenitic stainless steel is shown in Fig. 1. It includes the following stages (3,4):

- examining the sample and choosing a place to cut the sample for research;
- cutting the sample with constant cooling;
- preparation of metallographic samples by pouring the samples with epoxy resin in special molds;
- consecutive grinding of the examined surfaces with sandpapers of different abrasive sizes;

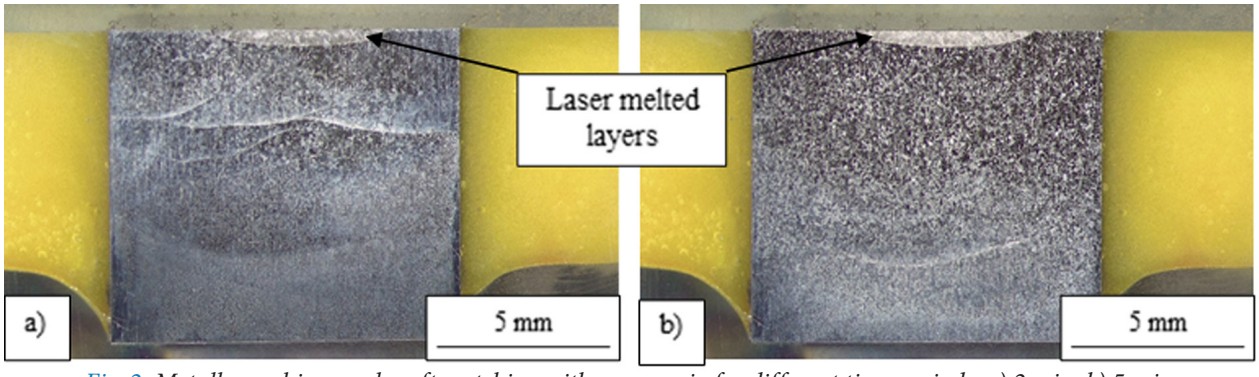


Fig. 2. Metallographic samples after etching with aqua regia for different time periods: a) 2 min; b) 5 min.

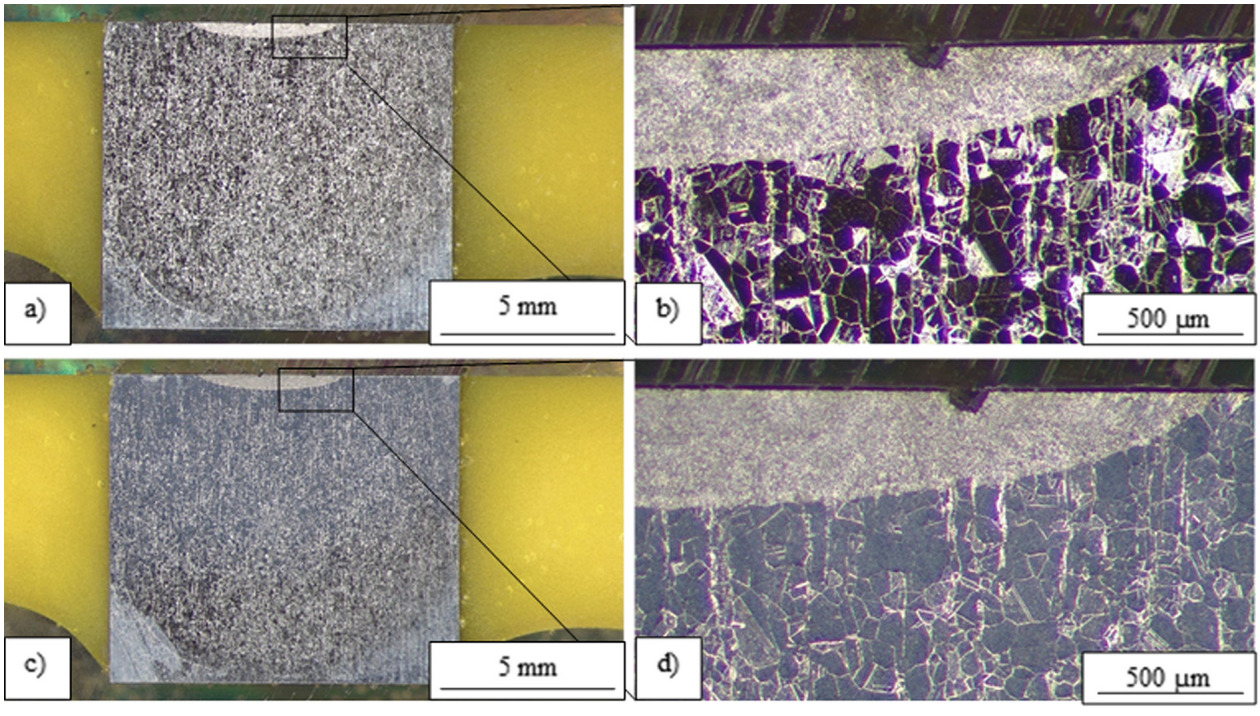


Fig. 3. Metallographic samples after 5 min of etching with aqua regia: a) and b), followed by 5 s of polishing: c) and d).

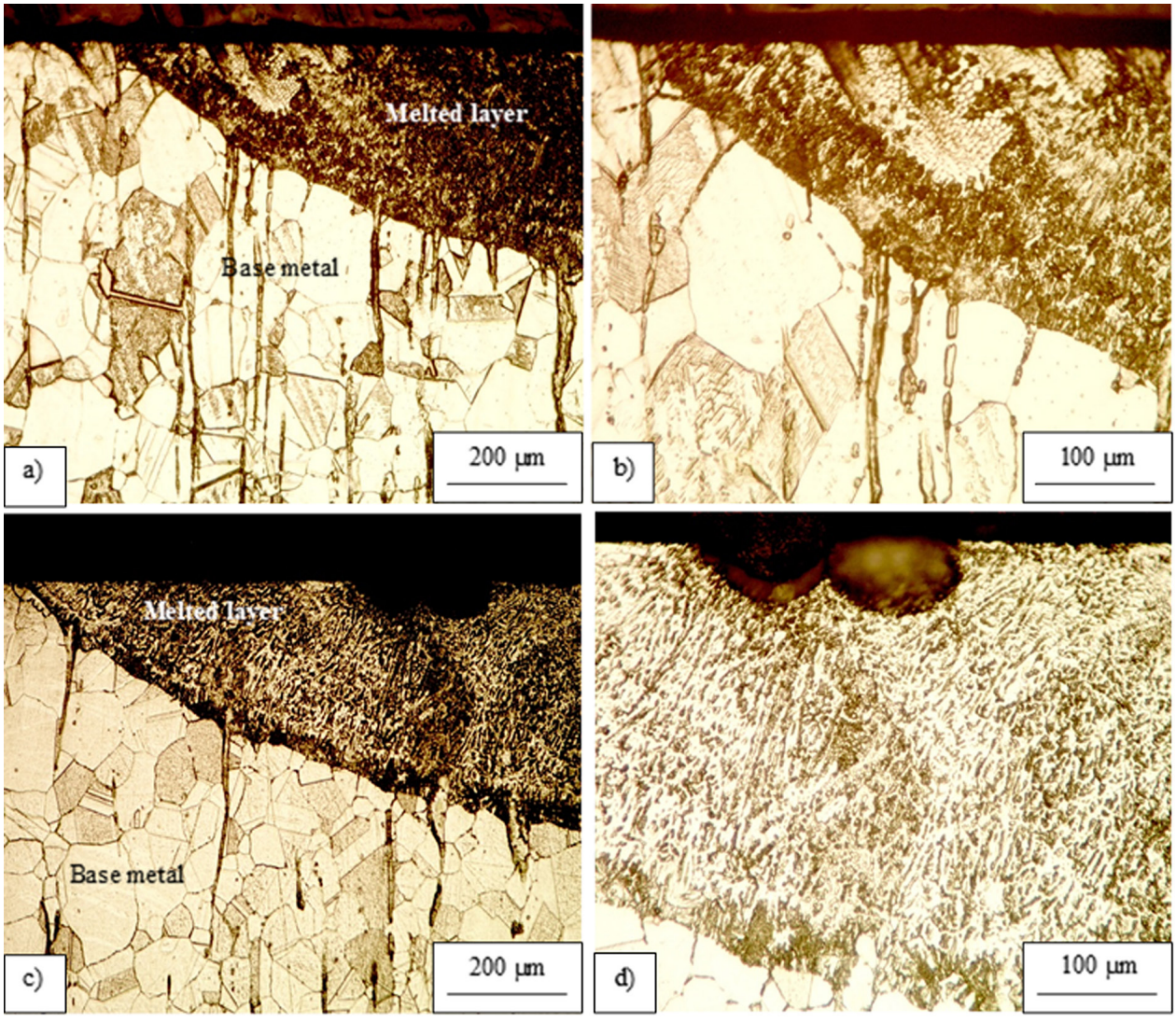


Fig. 4. Microstructure of base metal and laser-melted layer after 5 min of etching: a) and b), followed by 5 s of polishing: c) and d).

- polishing to mirror smoothness using various discs and suspensions;
- etching of the microstructure with reagents;
- observation of the microstructure with optical or scanning electron microscopes.

According to the ASTM E407-99 standard (5), when working with aqua regia, the microstructure is etched in 5–60 s by dipping the specimen or smearing with a swab. According to Vander Voort GF et al. (4), using a swab results in a more uniform etching of the microstructure.

In our work, a swab was used and, after etching for 30 s, no clear boundaries of the structural elements were observed. Processing for 2 min gave a satisfactory result, and the best display of the microstructure was obtained after etching for 5 min (Fig. 2). It can be seen in Fig. 2a that the microstructure of the laser-melted layer was clearly manifested after a treatment of 2 min, but the microstructure of the base metal was not well developed. Therefore, the treatment time with the reagent was prolonged. After etching for 5 min, all elements of the microstructure of the base metal were observed very well (Fig. 3 and Fig. 4). Austenite grain boundaries, twinning of some of them, δ-ferrite in shape of stripes, and a small amount of spheroidal carbides along the grain boundaries were clearly visible (Fig. 3a,b and

Fig. 4a,b). However, the microstructure of the laser-melted layer was over-etched and the individual elements could not be clearly distinguished. Therefore, the samples were further polished for no more than 5 s, which resulted in weaker contours of the microstructure of the base metal (Fig. 3c,d) but better visibility of details in the molten layer (Fig. 4c,d) .

DISCUSSION

The conventional procedure for manual preparation of metallographic samples includes mainly 3 processes: cutting the sample, successive grinding with several types of sandpaper, and a 3-stage polishing with abrasive suspensions of different sizes (Table 1). When using modern equipment, grinding can be done in 1 stage, followed by polishing (4). Electrolytic polishing is most suitable for austenitic stainless steels. These steels are relatively easy to polish, but the manifestation of the microstructure is significantly more difficult due to the variety of structural elements with different etching degrees. During laser treatment, the microstructure of the surface layer changes, which makes it even more difficult to study together with the microstructure of the base metal. Using standard reagents to etch austenitic stainless steels to reveal grain boundaries is relatively difficult. Since the surface of the austenitic steels is passivated, it is necessary to create reduction conditions. Therefore, the reagents for these purposes contain hydrochloric, sulfuric, and hydrofluoric acids. Nitric acid can be used alone, but most often it is used together with hydrochloric acid in a ratio of 3/1 (HCl/HNO_3), i.e., aqua regia (4,5). Since the individual phases of the microstructure exhibit different etching tendencies, some authors propose sequential etching procedures for their identification.

On this basis, since the microstructure of the laser-melted layer of the studied steel exhibits a stronger reaction to aqua regia, it is necessary to carry out the etching procedure in two successive stages. The first stage has a duration of 2 min to reveal the microstructure of the laser-melted layer. After the necessary metallographic studies are done, proceed with the second stage—an additional etching of 3 min to develop and study the microstructure of the base metal. Based on this analysis, an optimized procedure for developing and studying the microstructures of the base metal and laser-melted layer of AISI 321 austenitic stainless steel is proposed and shown in Fig. 1.

CONCLUSION

The methodology for preparation of metallographic samples has been optimized in the present paper in order to study the microstructure of a laser-melted layer and of a base metal in the same sample. For this purpose, samples of AISI 321 austenitic stainless steel with laser-melted surface layers were ground and polished by a conventional procedure, but etched with aqua regia for different times. It was found that:

- The microstructure of the laser-melted layers is revealed after a shorter exposure time (2 min) of the reagent. Longer exposure time leads to over-etching and unclear structural elements.
- A longer exposure time (5 min) is required to reveal the microstructure of the base metal. Subsequent polishing of the sample results in more obscure structural features.

An optimized procedure is proposed in which the etching process is carried out in two consecutive stages. In the first stage, the microstructure of the laser-melted layer is revealed in 2 min exposure time. After the necessary metallographic studies are done, the second stage continues—an additional etching of 3 min to reveal and study the microstructure of the base metal.

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