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Robert Kosturek

robert.kosturek@wat.edu.pl

Military University of Technology Faculty of Mechanical Engineering: Wojskowa Akademia Techniczna
im Jaroslawa Dabrowskiego Wydział Inżynierii Mechanicznej <https://orcid.org/0000-0002-6663-0529>

Janusz Torzewski

Krzysztof Grzelak

Tomasz Ślęzak

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Post-weld heat treatment of AA7075 friction stir welded joints manufactured under various tool rotation speeds and cooling conditions

Robert Kosturek*, Janusz Torzewski, Krzysztof Grzelak, Tomasz Ślęzak

¹ Military University of Technology, Faculty of Mechanical Engineering, 2 gen. S. Kaliskiego str., 00-908 Warsaw, Poland

* *robert.kosturek@wat.edu.pl*

Abstract: This study investigated the effect of post-weld heat treatment (PWHT) on the microstructure, microhardness, tensile properties, and fracture behavior of friction stir welded AA7075 joints produced under different cooling conditions and tool rotational speeds. Joints were welded in air, under water, in quenching oil, and with a cooling fluid, and were subsequently solution treated and artificially aged. PWHT increased and homogenized microhardness, with the stir zone generally exhibiting higher hardness than the surrounding regions. Abnormal grain growth (AGG) occurred in the shoulder-driven zone of all specimens, irrespective of cooling condition or tool rotational speed, and was also observed in the pin-driven zone of most joints. For defect-free specimens, PWHT increased yield strength by more than 70% and ultimate tensile strength by approximately 35%, with the highest UTS values exceeding that of the base material. The air-welded joint also showed nearly doubled ductility and was the only heat-treated joint to fracture outside the weld region. Defective joints exhibited limited ductility. Fractography revealed intergranular cracking, dimpled transgranular fracture, particle-assisted microvoid formation, and strain localization. Overall, PWHT improved hardness and strength but did not prevent AGG, particularly in the shoulder-driven zone.

Keywords: friction stir welding, aluminum, mechanical properties, heat treatment, welding parameters

1. Introduction

In recent years, many studies have focused on improving the mechanical properties of FSW joints made of precipitation-hardened aluminum alloys [1-3]. Among the numerous approaches considered, post-weld heat treatment (PWHT) deserves particular attention due to its greatest potential for restoring the strength of the welded alloy, as well as the difficulties it unfortunately poses in the case of FSW joints [2,4,5]. In principle, the two-stage heat treatment consisting of solution treatment and artificial aging makes it possible to obtain a uniform distribution of the strengthening phase throughout the entire volume of the joint, and this approach is commonly applied to joints of 2XXX and 6XXX aluminum alloys welds by conventional means [6-8]. In the case of FSW joints, precipitation-hardening heat treatment has a highly unfavorable side effect in the form of abnormal grain growth (AGG) occurring in the stir zone (SZ) [2,7]. This phenomenon has been reported in a number of research studies, including those associated with NASA's development of friction stir welded and spin-formed Al-Li 2195 structures [9], where it was recognized as a major issue adversely affecting the mechanical performance of the weld region. Importantly, joints affected by AGG exhibit a uniform microhardness distribution, since precipitation strengthening in aluminum alloys is far more effective than grain-boundary strengthening, as well as favorable mechanical properties in the static tensile test [10]. However,

as demonstrated in studies conducted by the authors the same joints, when subjected to low-cycle fatigue testing, showed a significant deterioration in fatigue life compared with their non-heat-treated counterparts. It was clearly demonstrated that the coarse-grained structure formed during heat treatment, together with the presence of porosity along the grain boundaries, promotes the initiation and propagation of fatigue cracks in heat-treated FSW joints [2]. The potential benefits that heat treatment without the formation of a coarse-grained structure could offer for FSW joints made of precipitation-hardened aluminum alloys continue to drive research aimed at avoiding, or at least limiting, AGG in the SZ. In this context, the identification of the key factors responsible for AGG has played an important role in shaping research strategies focused on its prevention. According to Chen and Russell [11], AGG in friction stir welded Al-Li 2195 arises from the microstructural instability of the fine-grained SZ during post-weld solution heat treatment. They further showed that AGG is promoted by the dissolution of soluble precipitates, which reduces grain-boundary pinning, and that its development depends strongly on both the level of prior plastic deformation and the heating rate to the solution treatment temperature. This interpretation was later further developed by Kalinenko et al. [4], who, in their study on friction stir welded AA6061-T6, demonstrated that AGG may initiate already during heating to the solution treatment temperature, thereby pointing to the intrinsic thermal instability of the fine-grained stir-zone microstructure.

At the same time, a number of studies have appeared in recent years reporting various approaches to mitigating AGG during the PWHT of FSW joints in the precipitation-hardened aluminum alloys. One of the approaches is to increase the fraction of secondary-phase particles in the SZ, thereby enhancing grain-boundary pinning [12,13]. Zou et al. [12] demonstrated that AGG in the SZ of FSW AA6061 can be effectively suppressed by promoting the in situ formation of intermetallic particles during welding, since the introduction of ball-milled Al-Ti powder mixtures led to the formation of secondary-phase particles, including Al_3Ti , which increased particle density and size, enhanced grain-boundary pinning, and ultimately stabilized the microstructure during PWHT. An increased fraction of secondary-phase particles in the SZ can be achieved not only by introducing powder during welding, but also by applying underwater cooling; in this context, Ghazaei Najafabadi et al. [13] reported for underwater FSW (UWFSW) AA2024/6061 lap joints that water cooling increased the precipitate volume fraction in the SZ by 36%, thereby enhancing grain-boundary pinning and markedly restricting grain growth during PWHT. Another noteworthy strategy for mitigating AGG is post-process plastic deformation prior to heat treatment, as demonstrated by Vysotskiy et al. [5] for friction-stir welded AA6061, where pre-straining before PWHT was shown to suppress AGG, with the effectiveness of this approach depending on both the strain level and the strain path relative to the welding direction. According to some reports, AGG can also be mitigated by controlling the parameters of the FSW process itself; for example, Panwariya and Dwivedi [14] showed for friction-stir welded AA7075-T651 that variation in tool rotational speed significantly affected the susceptibility to AGG during post-weld T6 treatment, with higher rotational speeds promoting a more homogeneous stir-zone microstructure and a higher fraction of high-angle grain boundaries, whereas lower rotational speeds were associated with a boundary-character distribution more prone to discontinuous grain growth.

Among all the approaches analyzed here, particular attention should be paid to the application of additional water cooling and the control of tool rotational speed, as neither of these methods requires the problematic introduction of powder into the welding zone nor additional plastic deformation prior to heat treatment. In this context, the present paper constitutes a continuation of the authors' previous studies on the effect of process parameters on the properties of FSW and submerged FSW (SFSW) joints in the AA7075-T651 alloy [15,16]. Despite the increasing research interest in PWHT of friction stir welded precipitation-hardened aluminum alloys, this issue remains insufficiently explored and still requires systematic experimental verification, particularly with regard to the effectiveness of individual strategies for mitigating AGG. In the present study, the primary objective was to verify whether increasing the tool rotational speed and applying additional cooling in FSW can significantly limit grain growth during PWHT.

2. Materials and methods

The investigated material was AA7075 alloy in the form of 5 mm thick sheets. Two different batches of this alloy were used in the study: one in the T651 temper (supplied by BIKAR-METALLE GmbH) and the other in the T6 temper (supplied by AMAG rolling GmbH). The chemical composition and the basic mechanical properties of the materials used are summarized in Tables 1 and 2 below.

Tab. 1. Properties of used AA7075-T651 alloy.

Chemical composition								
Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
0.071	0.122	1.610	0.025	2.596	0.197	5.689	0.041	Base
Mechanical properties								
Microhardness		YS		UTS		A		
170 HV0.1		548 MPa		584 MPa		14 %		

Tab. 2. Properties of used AA7075-T6 alloy.

Chemical composition									
Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Ti + Zr	Al
0.06	0.12	1.6	0.03	2.7	0.18	5.9	0.04	0.08	Base
Mechanical properties									
Microhardness		YS		UTS		A			
167 HV0.1		502 MPa		571 MPa		12 %			

The sheet edges were milled, while the top surfaces were abraded with sandpaper and cleaned with extraction gasoline immediately prior to the joining process. Butt-joint welding of the sheets was performed on an ESAB Legio 4 UT machine using an ESAB tool with catalog number 0810134-001, a tilt angle of 2°, and a plunge depth of -4.8 mm. FSW joints were

produced using different cooling media and tool rotational speeds, and the complete set of processing parameters together with the corresponding sample designations is summarized in Table 3. The joints were produced perpendicular to the rolling direction of the sheets. For submerged FSW (SFSW) joints, the distance between the liquid surface and the top surface of the joined sheets was maintained at 20 mm.

Tab. 3. Designation and parameters of produced FSW joints.

Sample	Material	Welding velocity	Tool rotation speed	Cooling medium
Air	AA7075-T651	125 mm / min	600 rpm	Air (conventional FSW)
OH-70				OH-70 quenching oil
Water				Tap water
10%CF				9.8% Blasocut 2000 solution
600	AA7075-T6	100 mm / min	800 rpm	Air (conventional FSW)
800			1000 rpm	
1000			1200 rpm	
1200			1400 rpm	
1400				

The produced joints were subsequently subjected to heat treatment (T6) dedicated to the AA7075 alloy, consisting of solution treatment at 470°C for 2 h followed by aging at 120°C for 24 h (Figure 1). The heat treatment was carried out using a Nabertherm P300 furnace.

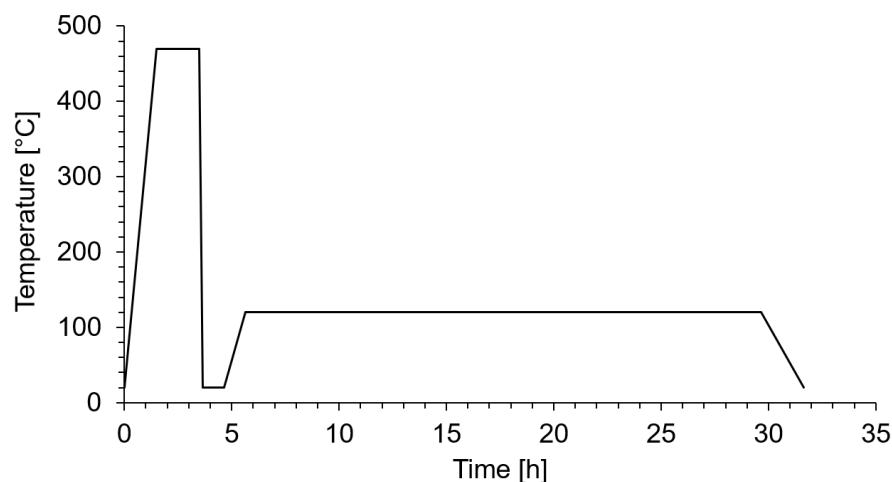


Fig. 1. Schematic illustration of the heat treatment procedure.

In the present study, heat-treated specimens are identified by the suffix “+HT” added to the sample designation. It should also be emphasized that the aim of this paper is not to analyze the effect of cooling media or FSW process parameters on joint properties, since these aspects have already been addressed in the literature [15,16], but rather to examine the behavior of the selected joints after post-weld heat treatment. Accordingly, the as-welded specimens are

included solely for reference purposes. Examination of the produced joints revealed that the 1200 and 1400 specimens contained void-type imperfections in the SZ. Nevertheless, these specimens were also subjected to heat treatment and included in the analysis, as the purpose of the study was not to obtain defect-free joints, but to assess the relationship between tool rotational speed and the susceptibility to AGG. Both the as-welded joints and those subjected to heat treatment were prepared using standard metallographic procedures, including resin mounting, grinding, and polishing. To reveal the microstructure, the specimens were etched with Keller's reagent. Microstructural observations were carried out using a Keyence VHX-7000 digital microscope. Further characterization included microhardness measurements, tensile testing, and fractographic analysis. The microhardness profiles of the fabricated joints were determined using a Struers DURA SCAN 70 hardness tester under a load of 0.98 N applied for 10 s. Measurements were taken along the weld cross-section at mid-thickness, i.e., 2.5 mm below the upper surface of the joined plates. The static tensile behavior of the joints was assessed in accordance with ASTM E8/E8M-13a using a 250 kN servo-hydraulic Instron 8802 MTL universal testing machine controlled by WaveMatrix software. Three specimens were tested for each condition, and the obtained results are presented as average values of yield strength (YS), ultimate tensile strength (UTS), and elongation at break (A). In addition, the fracture surfaces of the tested specimens were examined using a Jeol JSM-6610 scanning electron microscope.

3. Results and discussion

In the first stage of the macrostructural observations, the effect of tool rotation speed on the susceptibility to abnormal grain growth during post-weld heat treatment was analyzed. Selected macrostructural images are presented in Figure 2a–c below.

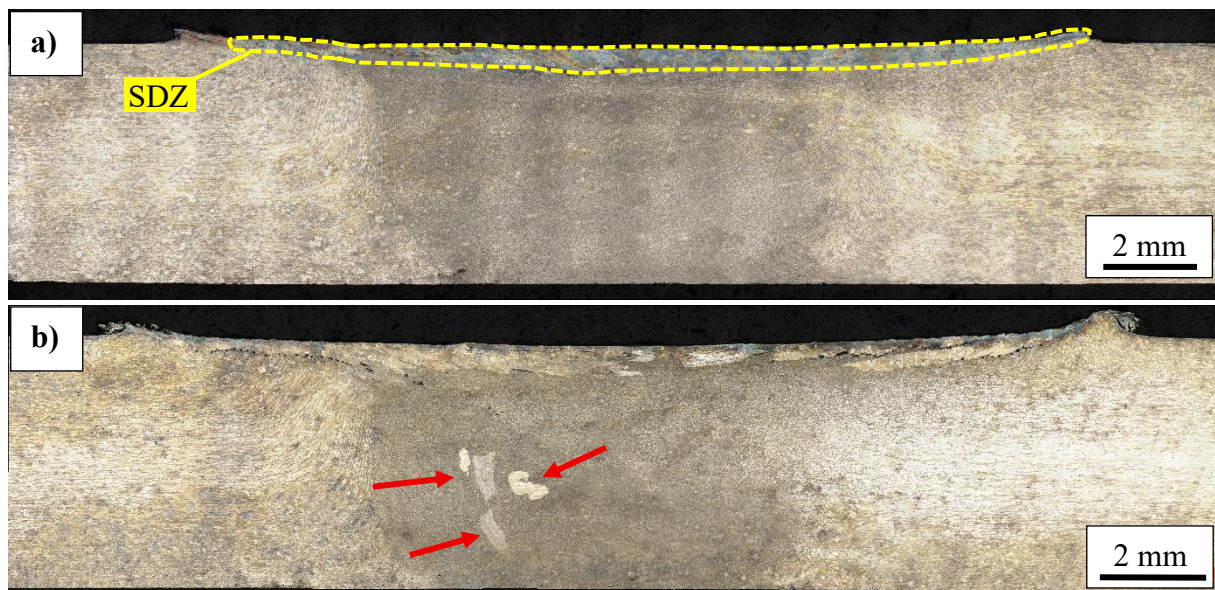




Fig. 2. Macrostructural images of selected samples: 600+HT (a), 1000+HT (b), 1400+HT (c).

Only the heat-treated specimens produced at tool rotational speeds of 600 rpm (Fig. 2a), 1000 rpm (Fig. 2b), and 1400 rpm (Fig. 2c) are shown in Figure 2, as they are sufficient to illustrate the trend identified in the present study. An important common feature is that, irrespective of the applied tool rotational speed, abnormal grain growth occurred in every specimen within the SZ formed by the action of the shoulder, i.e., the shoulder-driven zone (SDZ), as outlined for example in Fig. 2a. In contrast, no grain growth was observed in the pin-driven zone (PDZ) of the specimens produced at tool rotational speeds of 600 and 800 rpm. It was observed that the susceptibility to grain growth in the PDZ increased with increasing tool rotational speed, which can already be seen in the specimen welded at 1000 rpm (Fig. 2b). Approximately at mid-height of the joint, four grains about 500 μm in size were observed, shifted toward the advancing side with respect to the central part of the SZ (marked with red arrows in Fig. 2b). A further increase in tool rotational speed, apart from leading to the formation of joint imperfections, resulted in even more pronounced grain growth during post-weld heat treatment. As can be seen in the 1400+HT specimen, grain coarsening covered the same region as in the 1000+HT specimen (Fig. 2b), while additionally extending markedly into the lower part of the SZ (Fig. 2c). These results are inconsistent with the observations of Panwariya and Dwivedi [14], according to whom increasing the tool rotational speed in friction-stir welded AA7075-T651 reduced the tendency for AGG during post-weld T6 treatment by promoting a more homogeneous stir-zone microstructure and a higher fraction of high-angle grain boundaries. This highlights the lack of consensus regarding the effect of welding parameters on the susceptibility to AGG and underscores the need for further research in this area. Microstructural images of selected regions of the analyzed specimens are shown in Figure 3a–f.

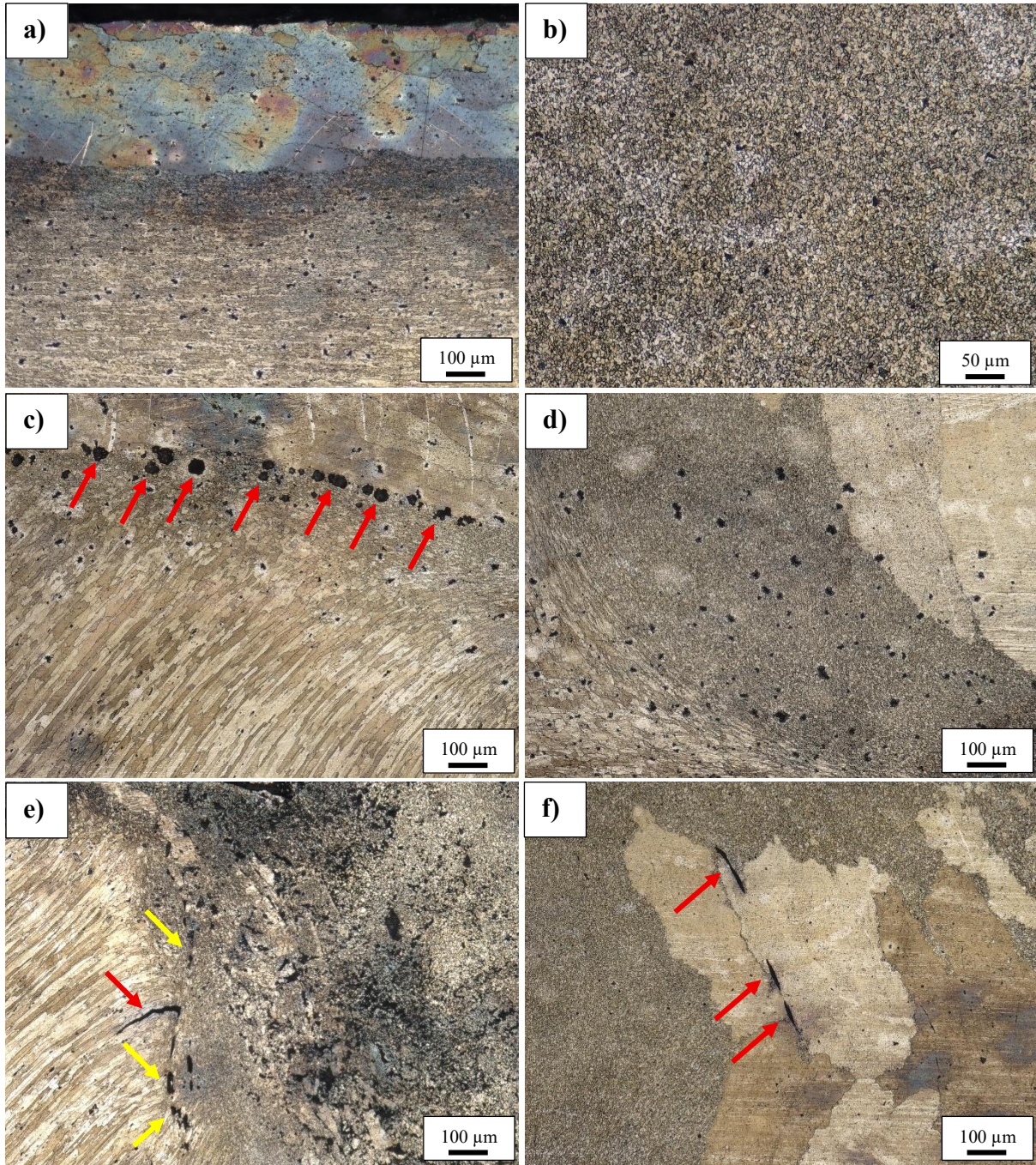


Fig. 3. Microstructural images of selected regions: SDZ (a) and PDZ (b) of 600+HT, upper part of TMAZ of 1000+HT (c), TMAZ/PDZ interface of 1200+HT (d) and 1400+HT (e), PDZ of 1400+HT (f).

The SDZ/PDZ interface in the 600+HT specimen is shown in Fig. 3a. A distinct boundary can be identified between the fine-grained PDZ microstructure and the coarse-grained SDZ. In some locations, the grain size in the SDZ exceeds 600 μm , which confirms the occurrence of AGG in this region (Fig. 3a). At the same time, the PDZ retained its fine-grained microstructure despite the applied heat treatment, with the average equiaxed grain size remaining at approximately 10 μm (Fig. 3b). This value is consistent with that reported in our previous study on the SZ microstructure of friction stir welded AA7075-T651 joints, in which the grain size was approximately 6 μm [15]. In some joints, heat treatment also introduced

additional defects, as can be observed in the 1000+HT specimen, where a band of pores with a size of approximately 30-40 μm formed at the TMAZ/coarse-grained SDZ boundary, located within a layer of fine grains (marked with red arrows in Fig. 3c). The region in which these heat-treatment-induced imperfections developed was described in detail in the as-welded condition in the authors' previous study [15], where plastic flow in the upper TMAZ/SZ region was found to drag grains between the SDZ and PDZ, causing severe compression, partial dynamic recrystallization, and ultimately the formation of an ultrafine-grained layer at the SDZ/PDZ interface. Even in the specimens where AGG developed in the PDZ, the outer layers of the SZ adjacent to the TMAZ retained a fine-grained structure, as illustrated by the 1200+HT specimen (Fig. 3d). At the same time, the regions in the 1200 and 1400 specimens where SZ imperfections had originally formed as a result of the excessively high tool rotational speed also preserved a fine-grained microstructure after heat treatment (Fig. 3e). However, new defects were observed there in the form of delamination of elongated grains (red arrow in Fig. 3e) and pores up to 50 μm in size located at the TMAZ/PDZ boundary (yellow arrows in Fig. 3e). In addition, some defects were found to occur directly within the PDZ in regions affected by AGG. A representative example is provided by the elongated pores located inside the enlarged grains in the PDZ of the 1400+HT specimen (marked with red arrows in Fig. 3f). This is consistent with observations made in previous studies on the post-weld heat treatment of FSW AA2519-T62 joints, where similar phenomena were also reported [2]. Macrostructural images of FSW joints produced using different cooling media and subsequently subjected to post-weld heat treatment are presented in Fig. 4a–f.

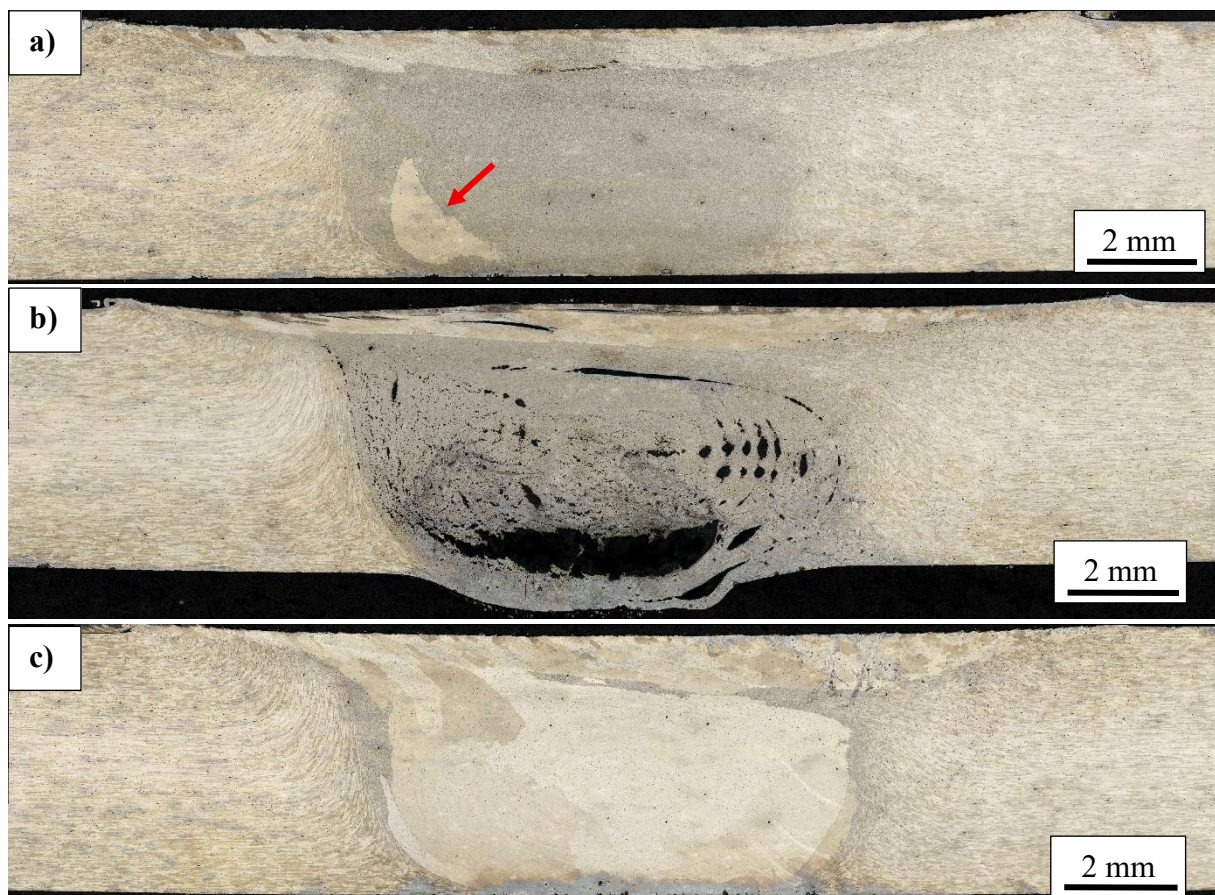




Fig. 4. Macrostructural images of selected samples: Air+HT (a), OH-70+HT (b), Water+HT (c), 10%CF+HT (d).

Similarly to the specimens produced at different tool rotational speeds, post-weld heat treatment also induced AGG in the SDZ in this case. Analysis of the macrostructure of the Air+HT specimen shows that, despite grain growth in the SDZ, the PDZ remained predominantly fine-grained, the only exception being a single large grain approximately 2 mm in size located in the lower part of the PDZ, close to the TMAZ on the retreating side (red arrow in Fig. 4a). Interestingly, the 600+HT (Fig. 3a) and Air+HT (Fig. 4a) specimens differ only in the applied welding speed (100 mm/min and 125 mm/min, respectively), as well as in the chemical composition (Tables 1–2) and temper condition of the alloy (T6 and T651, respectively). However, these seemingly subtle differences resulted in the occurrence of AGG in the PDZ of the Air+HT specimen. Although it is currently not possible to determine which of these factors was decisive for grain growth in the SZ, this observation is worth noting, as it illustrates how sensitive the PDZ is in terms of its susceptibility to grain growth during post-weld heat treatment. In the OH-70+HT specimen (Fig. 4b), although the PDZ retained its fine-grained structure, the applied heat treatment led to the formation of significant imperfections within the SZ. Numerous pores as well as clear delamination of individual material layers could be observed in the analyzed region. The arrangement of these imperfections corresponds to the successive layers of material mixed during joint formation; therefore, a possible explanation for the observed phenomenon is the thermal decomposition of quenching oil trapped in the material during the FSW process. Aluminum is characterized by high wettability by oil [17], and the short exposure time to FSW process temperatures (approximately 400°C) is likely insufficient to allow its evaporation from the edges of the joined elements, whereas prolonged exposure to the solution treatment temperature (470°C/2 h) may lead to its evaporation and potentially partial pyrolysis [18]. In addition, the thin oil film present between adjacent material layers likely contributed to weak bonding between them, which in turn facilitated their relatively easy separation during oil evaporation. Based on the observed macrostructure of the specimen, it may be concluded that the largest accumulation of quenching oil within the SZ was most likely located in its central lower part, close to the weld root. In both the Water+HT (Fig. 4c) and 10%CF+HT (Fig. 4d) specimens, AGG was observed throughout the predominant part of the PDZ. The only apparent exception seems to be the boundary regions between the TMAZ and SZ on both sides of the weld, which retained their fine-grained microstructure. These results are also inconsistent with literature reports, especially those of Ghazaei Najafabadi et al. [13], who found that underwater cooling in FSW AA2024/6061 lap joints increased the precipitate volume fraction in the SZ and significantly restricted grain growth during PWHT. By contrast,

in the present study, AGG was still observed in the predominant part of the PDZ, suggesting that the effectiveness of water-assisted cooling in suppressing grain growth during heat treatment is not universal and may depend on the specific alloy and welding conditions. Microstructural images of selected regions of the analyzed specimens are shown in Figure 5a–f.

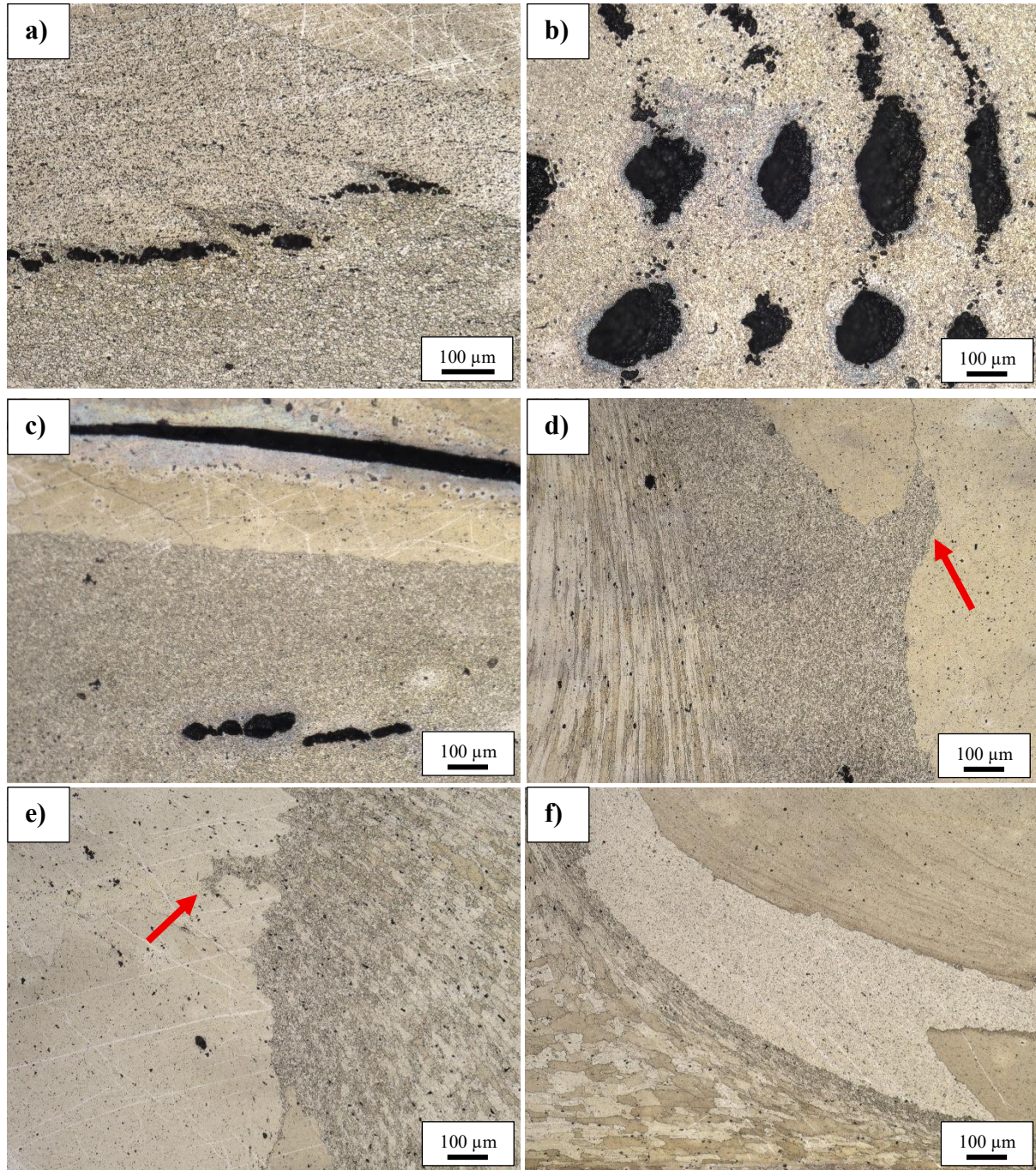
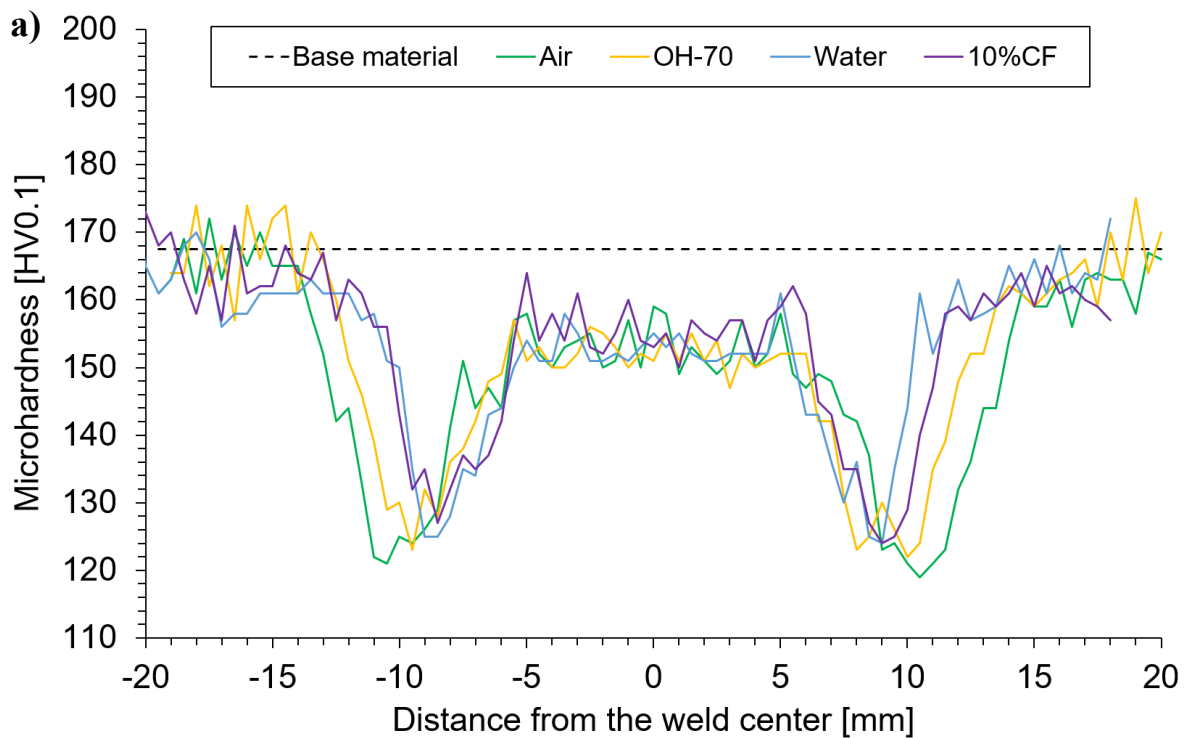


Fig. 5. Microstructural images of selected regions: SDZ/PDZ interface of Air+HT (a), PDZ (b) and SDZ/PDZ interface (c) of OH-70+HT, TMAZ/SZ interface of Water+HT at AS (d) and RS (e), lower part of TMAZ/SZ interface of 10%CF+HT.

The first image (Fig. 5a) shows a region of the Air+HT specimen in which the fine-grained PDZ is located directly beneath the coarse, enlarged grains of the SDZ, while also exhibiting imperfections formed during post-weld heat treatment. In this case, pore aggregates developed within the fine-grained layer adjacent to the SDZ, representing another example of defects of this type occurring at the interface between coarse-grained and fine-grained regions observed in the present study. The imperfections formed in the PDZ as a result of the decomposition of residual quenching oil are shown in Fig. 5b. They exhibit a rounded morphology, locally forming nearly circular pores with diameters exceeding 200 μm . In addition, the fine-grained microstructure of the welded material was observed to undergo noticeable deformation in the vicinity of these pores, which can be attributed to the pressure exerted by quenching-oil vapors during post-weld heat treatment. Similarly to the Air+HT specimen (Fig. 5a), pores were also observed near the PDZ/SDZ interface in the upper part of the OH-70+HT joint (Fig. 5c). The pores were located within the PDZ, approximately 500 μm from the SDZ boundary, and their arrangement appeared to form a continuation of the delamination line within the PDZ, visible on the retreating side in the macrostructural image (Fig. 4b). In addition, delamination of the enlarged grains was also observed in the SDZ, resulting in the formation of an elongated crack approximately 100 μm in width (Fig. 5c). In the Water+HT and 10%CF+HT specimens, AGG was observed throughout most of the PDZ, with only limited fine-grained regions retained in the outer layers adjacent to the TMAZ (Fig. 5d,e). They were also characterized by isolated fine-grained colonies extending approximately 100-200 μm into the coarse-grained region (red arrows in Fig. 5d,e). While the fine-grained region on the advancing side (Fig. 5d) exhibited predominantly equiaxed grains without a clearly defined deformation direction, the corresponding region on the retreating side (Fig. 5e) contained both fine dynamically recrystallized grains and larger plastically deformed grains representing remnants of the TMAZ. A similar coexistence of fine recrystallized grains and coarser deformed grains near the SZ/TMAZ boundary was reported by Mehri et al. [19], who attributed this heterogeneous microstructure to the concurrent operation of discontinuous and continuous dynamic recrystallization mechanisms. A residual band of fine grains, up to approximately 50 μm in width, could also be observed at the PDZ/TMAZ boundary in the lower part of the joint, separating the two regions (Fig. 5f). This region was also examined in the as-welded condition in the authors' previous study [15], where the material flow at the lower TMAZ/SZ boundary produced a layer of severely deformed and compressed grains surrounding the PDZ, approximately 100 μm thick, while higher heat input promoted partial dynamic recrystallization within this layer. The observations made in the present study are consistent with the findings of İpekoğlu et al. [20], who reported that, after PWHT of FSW AA7075 joints, narrow bands of fine-grained microstructure could remain adjacent to the TMAZ despite the occurrence of AGG in the neighboring stir zone. This corresponds well with the fine-grained regions retained at the PDZ/TMAZ and SDZ/PDZ interfaces in the present joints, confirming the strongly localized nature of microstructural stability within the SZ. The pronounced spatial variation in grain size observed in the investigated joints is consistent with the findings of Wang and Xu [21], who demonstrated for FSW AA2219 that AGG proceeds through different mechanisms in the SZ and TMAZ, with preferential growth in the SZ being promoted by locally advantageous grain size, low stored strain, and non-uniform particle pinning. The retention of narrow fine-grained

regions adjacent to abnormally coarsened areas may also be related to local differences in the initial weld microstructure and thermal history, as Lezaack and Simar [22] showed that the stability of thick 7XXX-series FSW joints during T6 heat treatment can be strongly modified through control of heat extraction during welding. Moreover, the asymmetric distribution of coarse grains toward the advancing side agrees with the observations of Ahmed and Wynne [23] for FSW AA2017, where PWHT-induced AGG developed mainly near the advancing side, while the retreating-side region largely retained its fine-grained structure, indicating that grain-growth susceptibility may also be texture dependent.

The microhardness profiles of the specimens produced using different cooling media are presented below for the as-welded condition [16] (Fig. 6a) and after heat treatment (Fig. 6b).



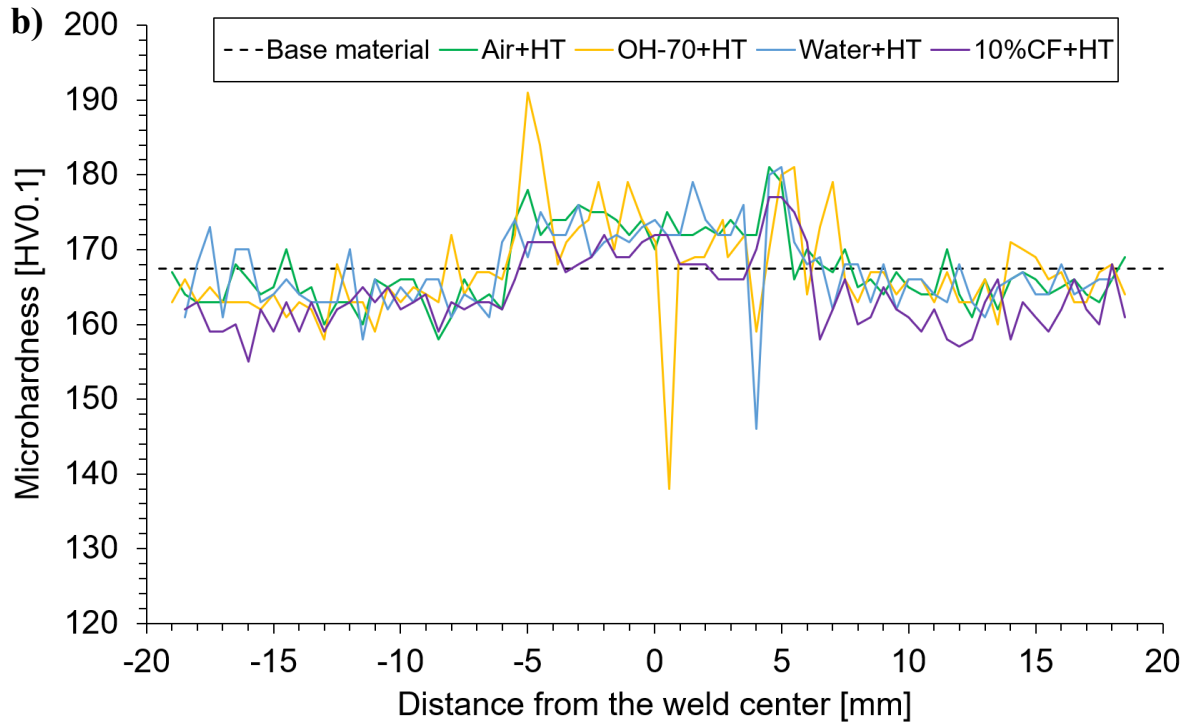


Fig. 6. Microhardness profiles of specimens produced using different cooling media: (a) as-welded condition [16] and (b) after post-weld heat treatment.

The effect of the cooling medium on the microhardness distribution of the investigated joints was described in detail in the authors' previous study [16] and is included here solely as a reference for assessing the influence of post-weld heat treatment on joint properties. In all cases, heat treatment (Fig. 6b) increased and homogenized the microhardness across the joint, indicating successful restoration of the strengthening precipitates. The slightly lower microhardness of the base material results from the different final temper condition: the initial material was supplied in the T651 temper, which includes plastic deformation prior to artificial aging, whereas after the applied heat treatment it corresponded to the T6 temper, characterized by a lower degree of strengthening. It is also noteworthy that elevated microhardness was observed within the SZ, including in the Water+HT and 10%CF+HT specimens, in which AGG was identified. The observation that grain growth did not lead to a reduction in microhardness in the heat-treated specimens, owing to the dominant contribution of precipitation strengthening, is consistent with the findings reported for post-weld heat-treated FSW AA2519 joints [2]. The only fluctuations, observed as locally reduced hardness values OF the OH-70+HT and Water+HT specimens, may be attributed to porosity present in the SZ and at the TMAZ/PDZ boundary.

The microhardness profiles of the specimens produced at different tool rotational speeds are presented below for the as-welded condition (Fig. 7a) and after heat treatment (Fig. 7b).

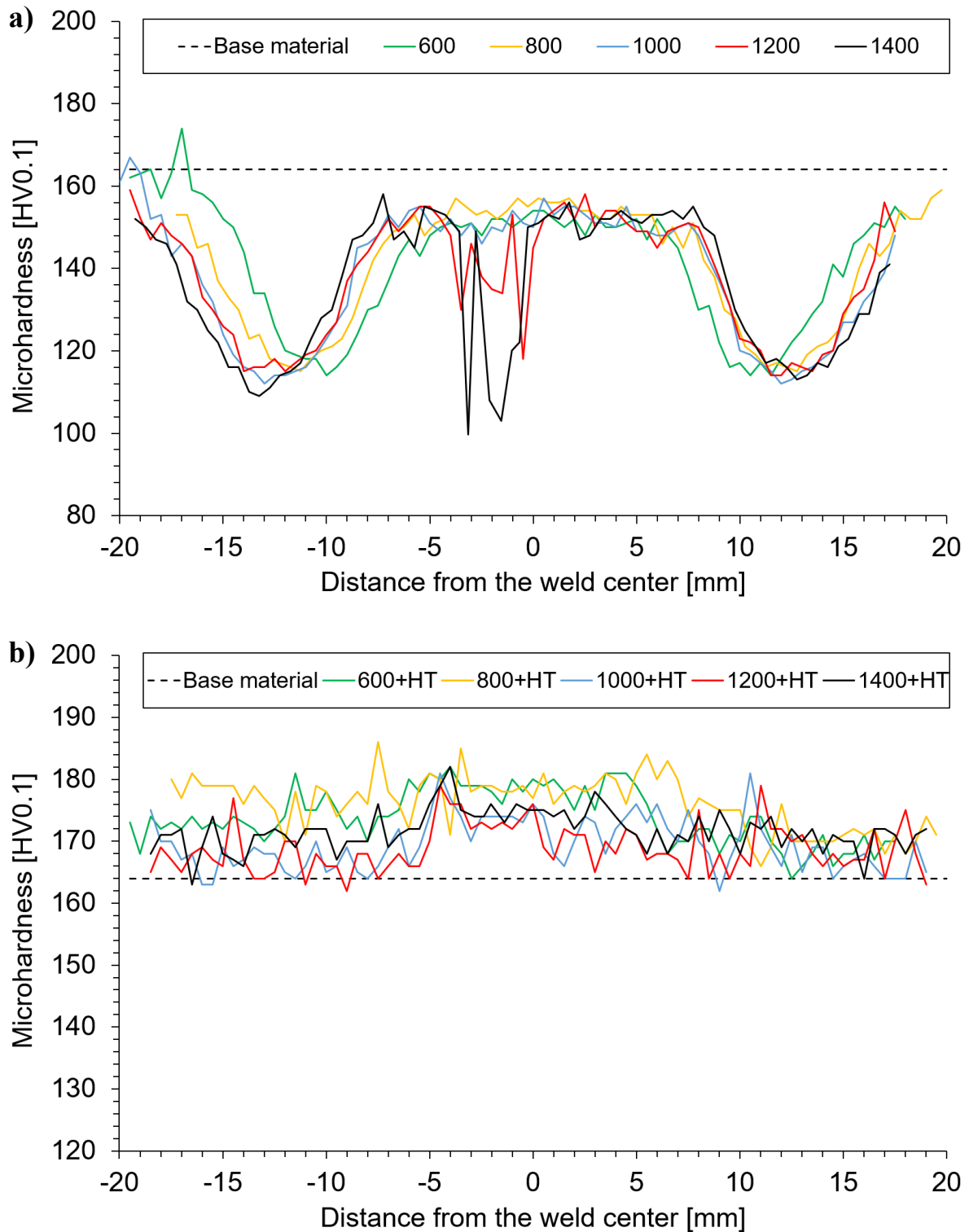


Fig. 7. Microhardness profiles of specimens produced at different tool rotational speeds: (a) as-welded condition and (b) after post-weld heat treatment.

The microhardness profiles obtained for the specimens produced at different tool rotational speeds (Fig. 7a) exhibited the characteristic W-shaped distribution typical of precipitation-hardened aluminum alloys. It can be observed that the HAZ broadened with increasing tool rotational speed, although the most pronounced change occurred between the

600 and 800 rpm specimens. At the same time, the hardness within the low-hardness region of all joints ranged from 108 to 114 HV0.1, with the highest value recorded for the 600 rpm specimen, which is consistent with literature reports in this respect [15,24]. Within the SZ, the hardness values showed some scatter and ranged from 144 to 156 HV0.1. Of particular note were the distinct regions of reduced hardness in the defective 1200 and 1400 rpm specimens, which should be associated with the presence of unbonded material regions within the stir zone, such as voids. As in the previous series of specimens, post-weld heat treatment increased and homogenized the microhardness across the investigated joints (Fig. 7b). Although some scatter was observed, the measured values were predominantly higher than those of the base material, confirming that the heat treatment was performed successfully. Interestingly, no regions of reduced hardness were detected within the SZ of the 1200 and 1400 specimens after heat treatment, despite the presence of pores in this area (Fig. 2), unlike in the OH-70+HT and Water+HT specimens (Fig. 6b). At the same time, all investigated specimens exhibited clearly higher microhardness within the SZ after heat treatment than in the remaining joint regions (Figs. 6b and 7b). The observations concerning the occurrence of AGG in the PDZ and SDZ, together with the increase in SZ microhardness, are summarized in Table 4.

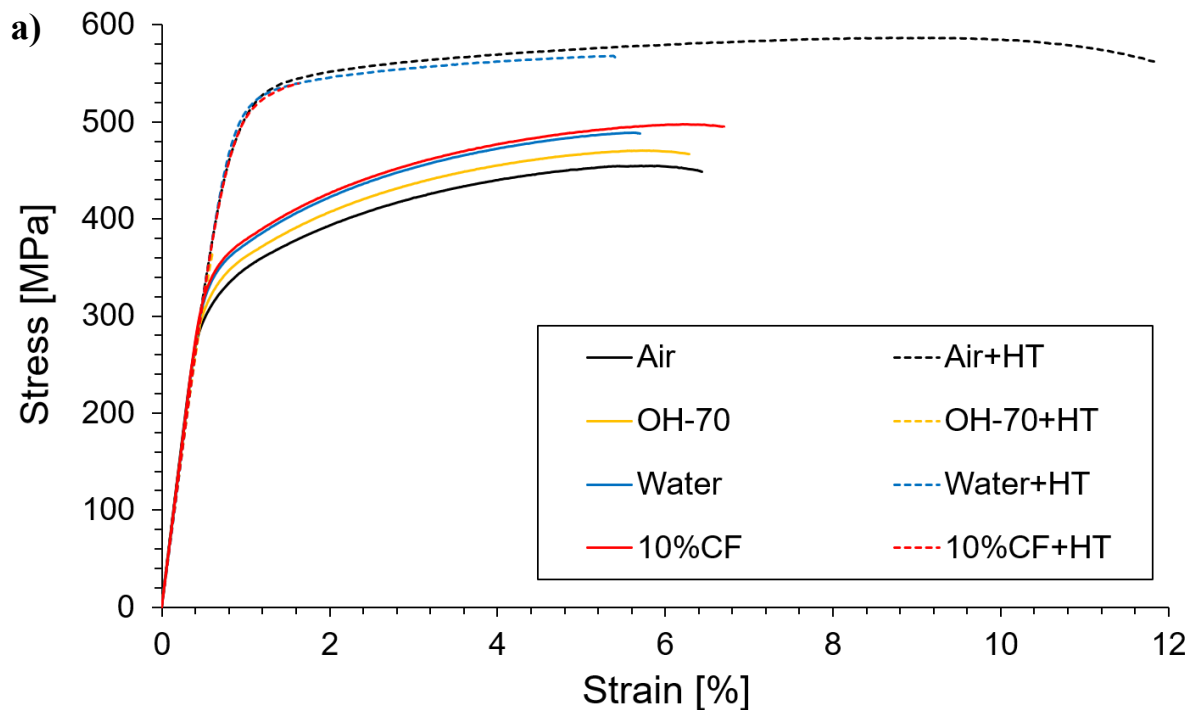
Tab. 4. Summary of AGG occurrence in the PDZ and SDZ and the corresponding increase in SZ microhardness after PWHT

Sample	Microhardness in SZ	AGG in SDZ	AGG in PDZ
Air+HT	+6.7%	Yes	Yes
OH-70+HT	+6.1%		No
Water+HT	+8%		Yes
10%CF+HT	+6.2%		
600+HT	+4.1%		Nie
800+HT	+3.4%		
1000+HT	+3.5%		Tak
1200+HT	+4.2%		
1400+HT	+4.1%		

The reported increases in microhardness refer to mean values and, given the observed scatter, do not allow for a reliable assessment of the exact increase achieved in each individual specimen. Therefore, the discussion was focused instead on the general cause of the elevated microhardness observed within the SZ. Because the increase in microhardness was observed in all specimens, irrespective of whether AGG occurred in the PDZ, it cannot be attributed to the possible retention of a fine-grained structure in this region. Instead, its origin should be sought in the precipitation-hardening response of the SZ. The intense plastic deformation at elevated temperature during FSW leads to the dissolution of strengthening η' precipitates in the stir zone [25], as well as to the fragmentation and redistribution of coarser equilibrium-phase particles [2]. Owing to their higher specific surface area and shorter diffusion distances, the fragmented

particles can dissolve completely during solution treatment, whereas coarse primary particles in the base material may remain partially undissolved, thereby limiting the actual supersaturation of the matrix. Since the density of precipitates formed during subsequent artificial aging has been shown to be proportional to the degree of matrix supersaturation [26], a denser and finer population of η' precipitates can develop in the SZ, resulting in higher hardness. A similar increase and homogenization of joint hardness after post weld cyclic solution treatment and aging, including elevated hardness within the SZ, was reported by Ramakrishna et al. [26] for FSW AA7075 joints.

Representative static tensile curves for the heat-treated specimens produced using different cooling media and tool rotational speeds are presented in Figs. 8a and 8b, respectively.



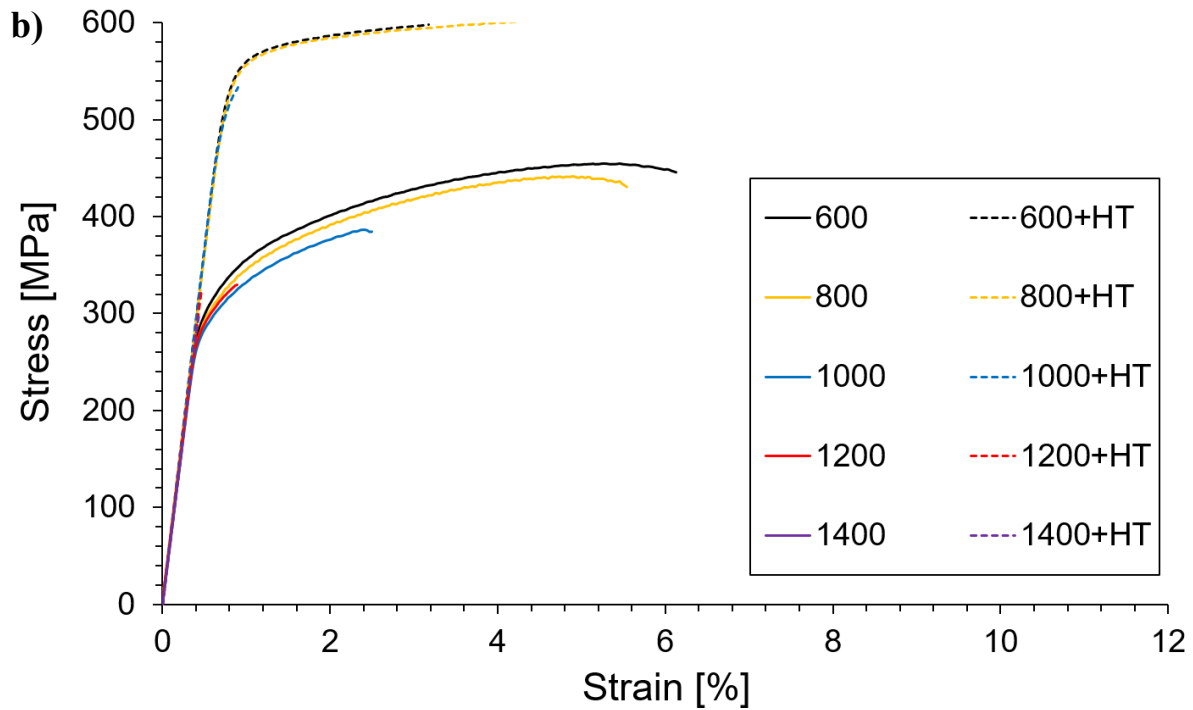


Fig. 8. Representative static tensile curves for specimens produced using (a) different cooling media [16] and (b) different tool rotational speeds, in the as-welded and heat-treated conditions.

For the specimens produced using different cooling media, heat treatment affected the mechanical properties of the individual joints to varying degrees (Fig. 8a). The Air+HT and Water+HT specimens exhibited substantially higher yield strength and ultimate tensile strength, while the former also showed an almost twofold increase in ductility, bringing its mechanical properties closer to those of the base material. Despite the marked improvement in strength, the 10%CF+HT specimen exhibited a considerable reduction in ductility, with total elongation to fracture not exceeding 2%. As expected, the heavily defective OH-70+HT specimen fractured at approximately 350 MPa and reached an elongation of less than 1%. The specimens produced at different tool rotational speeds exhibited an inverse relationship between the applied rotational speed and both their strength parameters and ductility (Fig. 8b). In this case, the applied post-weld heat treatment increased the strength parameters of the specimens produced at the lowest tool rotational speeds (600+HT and 800+HT), whereas a substantial reduction in ductility was observed for the remaining conditions. It should be emphasized that, owing to the presence of defects, the 1200 and 1400 specimens already exhibited relatively low UTS values and very limited ductility in the as-welded condition. Heat treatment did not significantly alter their mechanical performance, indicating that their strength was governed primarily by joint imperfections rather than by the temper condition.

The mean values and standard deviations of the basic mechanical properties (YS, UTS) of the specimens produced using different cooling media are presented below for the as-welded condition [16] (Fig. 9a) and after post-weld heat treatment (Fig. 9b).

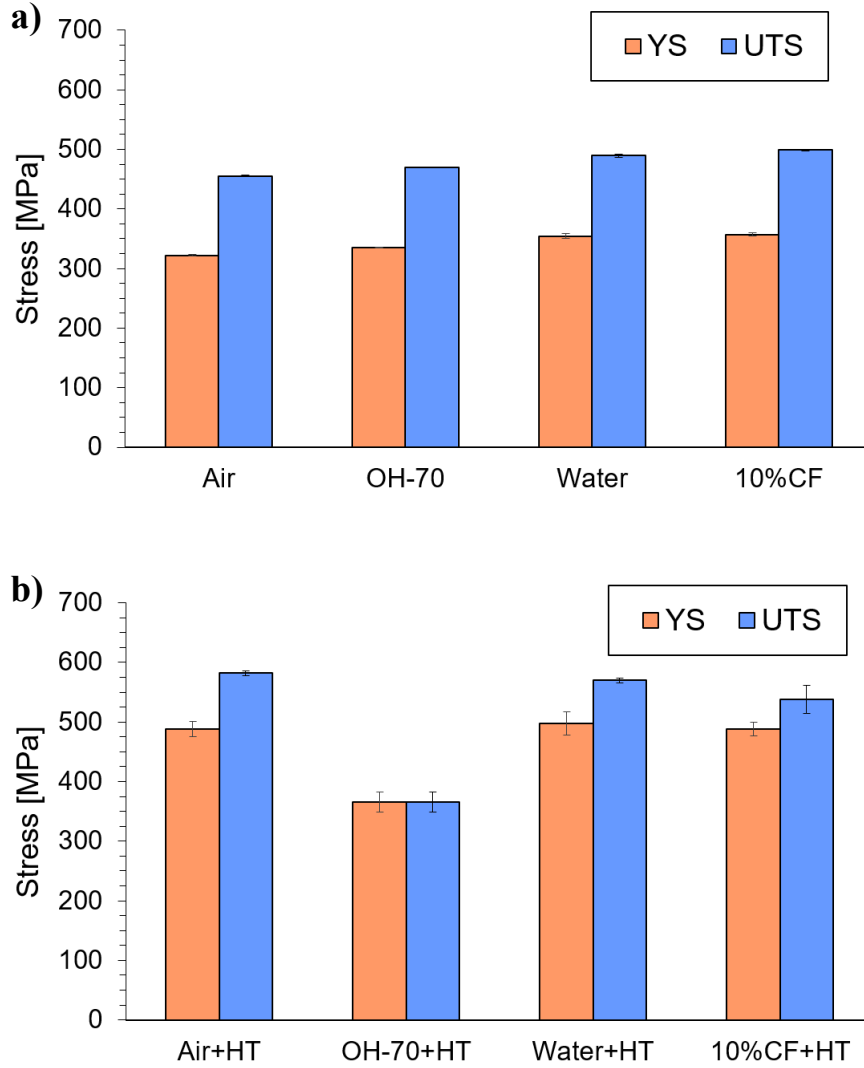


Fig. 9. Mean values and standard deviations of the basic mechanical properties of specimens produced using different cooling media: (a) as-welded condition [16] and (b) after post-weld heat treatment.

For the specimens produced using different cooling media, the recorded tensile strength was directly proportional to the amount of heat absorbed by the cooling medium during welding, as discussed in detail in [16]. As can be seen, post-weld heat treatment improved the strength properties to varying degrees, primarily by increasing the yield strength by approximately 40–50%. The only exception to this trend was the OH-70+HT specimen, owing to the substantial number of heat-treatment-induced imperfections in the PDZ (Fig. 4b). The UTS values of the Air+HT, Water+HT, and 10%CF+HT specimens exceeded 535 MPa, corresponding to a joint efficiency of approximately 91%. The greatest increase in UTS was recorded for the air-welded joint, whose relatively low as-welded UTS of 455 MPa provided the greatest potential for strengthening. By contrast, the specimen produced using the cooling lubricant exhibited only an 8% increase in UTS, from 500 MPa in the as-welded condition to 538 MPa after heat treatment. It should also be noted that the heat-treated specimens showed markedly greater scatter in both YS and UTS, indicating substantially lower repeatability of

mechanical properties than in the as-welded condition. Analogous plots were prepared for the specimens produced at different tool rotational speed (Fig. 10).

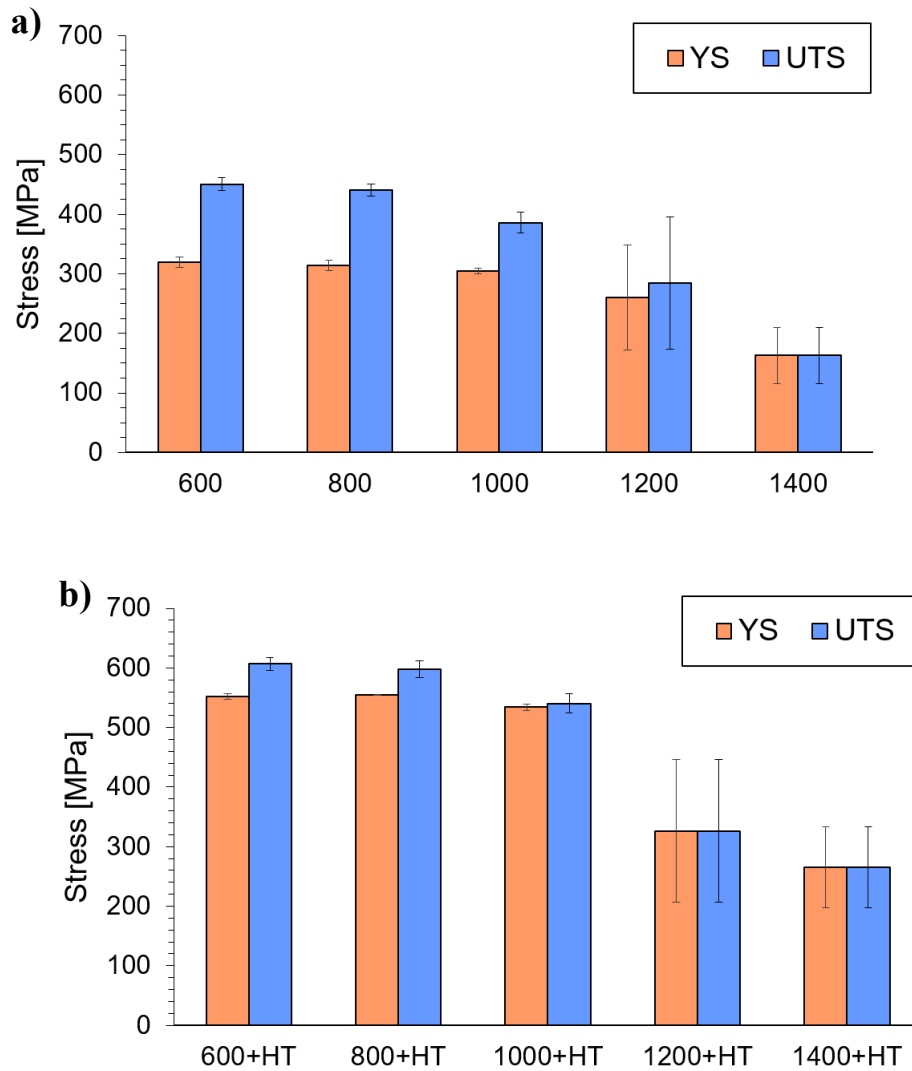


Fig. 10. Mean values and standard deviations of the basic mechanical properties of specimens produced at different tool rotational speeds: (a) as-welded condition and (b) after post-weld heat treatment.

The as-welded specimens clearly illustrate the relationship between tool rotational speed and the resulting YS and UTS values (Fig. 10a). The 1200 and 1400 rpm specimens may be excluded from this comparison owing to the presence of imperfections and the resulting substantial scatter in the reported mechanical properties. For the 600, 800, and 1000 rpm specimens, the yield strength remained very similar at approximately 310 MPa, whereas the greatest differences were observed in UTS, which decreased markedly with increasing tool rotational speed. As in the previous specimen series, the applied heat treatment generally increased the basic strength parameters of the investigated joints (Fig. 10b). For the defect-free joints, increases of more than 70% in YS and approximately 35% in UTS were recorded,

resulting in UTS values of 607 MPa for 600+HT, 597 MPa for 800+HT, and 540 MPa for 1000+HT. In the first two cases, these values exceeded the UTS of the base material (571 MPa). Even for the 1200+HT and 1400+HT specimens, increases in the mean strength parameters were recorded, although the UTS values remained clearly limited by the substantial presence of PDZ imperfections introduced during welding. The mean percentage changes in YS and UTS after PWHT are summarized in Table 4.

Tab. 4. Percentage changes in YS and UTS after PWHT.

Sample	YS after PWHT	UTS after PWHT
Air	+51%	+27%
OH-70	+9%	-22%
Water	+40%	+16%
10%CF	+37%	+8%
600	+72%	+35%
800	+76%	+36%
1000	+75%	+40%
1200	+25%	+15%
1400	+63%	+63%

To relate the observed changes in mechanical properties to the available literature, the study by Ramakrishna et al. [27] may be taken as a useful reference point, as the authors reported that cyclic solution treatment followed by artificial aging of FSW AA7075 joints increased UTS by 3% and YS by 25%, while reducing ductility by approximately half and shifting the fracture location from the HAZ to the SZ. The same general trends are consistent with the observations reported in that study (Fig. 8); however, the increases in strength obtained in the present work were substantially greater (Table 4). The Air+HT specimen (Fig. 8a) is particularly noteworthy, as it exhibited not only pronounced increases in YS and UTS, but also an almost twofold improvement in ductility and was the only heat-treated specimen to fracture outside the weld region. The changes in mechanical properties observed in the present study are also consistent with the results reported by İpekoğlu et al. [20], who showed that PWHT increased the YS and UTS of FSW AA7075-T6 joints to approximately 480–500 MPa and 500–510 MPa, respectively, corresponding to a tensile joint efficiency of up to 97%. Despite this substantial strength recovery, elongation remained at only about 2%, which the authors attributed to the heterogeneous post-heat-treatment microstructure and preferential fracture along the interface between AGG regions and retained fine-grained bands. Panwariya et al. [28], in turn, investigated the effects of stationary-shoulder FSW (SSFSW) and conventional-shoulder FSW (CSFSW) on susceptibility to AGG during post-weld heat treatment, while also reporting the corresponding mechanical properties. Irrespective of the shoulder configuration, UTS increased by approximately 40% to about 500 MPa, accompanied by a substantial reduction in ductility. Although AGG occurred in the CSFSW specimen but was suppressed in

the SSFSW specimen, both heat-treated joints exhibited nearly identical mechanical properties. This finding broadly corresponds to the observations made in the present study, which indicate that there is no straightforward relationship between the occurrence of AGG and the mechanical properties determined in static tensile testing, including ductility. This is illustrated by the Air+HT specimen, which exhibited one of the lowest extents of AGG in the PDZ, and the 600+HT specimen, in which no AGG was observed in the PDZ, yet their elongations to fracture differed by nearly a factor of four, amounting to 12% and 3%, respectively. This differs from the findings of İpekoğlu et al. [20], who linked the limited ductility of PWHT-treated AA7075 joints directly to fracture along the interface between AGG and retained fine-grained regions, further indicating that the influence of AGG on tensile behavior is complex and requires additional systematic investigation.

In the final stage of the study, a microfractographic analysis of the fracture surfaces was performed. FSW joints produced in air at a tool rotational speed of 600 rpm fractured outside the weld, within the HAZ on either the advancing or retreating side. Increasing the tool rotational speed to 1000 rpm shifted the fracture location toward the central region of the weld, although the fracture path remained slightly offset from the weld axis toward either the advancing or retreating side (Fig. 11). This change in fracture location is consistent with the tensile-test results, which showed a pronounced decrease in UTS with increasing tool rotational speed.



Fig. 11. Fracture locations of FSW joints produced in air at tool rotational speeds of 1000 rpm after static tensile testing.

The fracture region exhibited a markedly refined grain structure, with crack propagation occurring predominantly along grain boundaries and through coarse second-phase particles (Fig. 12).

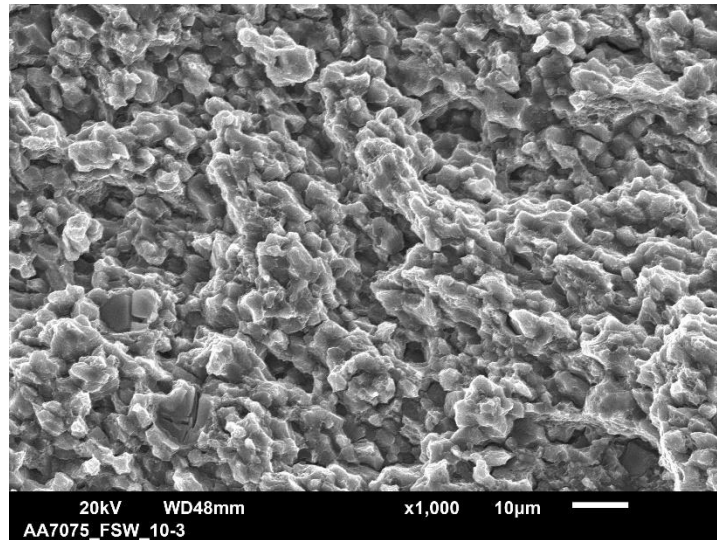


Fig. 12. Fracture surface of the joint produced at 1000 rpm.

As demonstrated in the preceding section, post-weld heat treatment generally led to a significant improvement in the mechanical properties; however, it also altered the fracture behavior. Irrespective of the tool rotational speed, all heat-treated specimens fractured within the central region of the weld, with the fracture path slightly offset from the weld axis toward the retreating side (Fig. 13).



Fig. 13. Fracture location of the 600+HT specimen after static tensile testing.

Microscopic examination of the fracture surfaces revealed subsurface cavities in both specimens, located at a depth of approximately 0.5 mm, with a greater number observed in the specimen produced at the higher tool rotational speed. Representative examples of the observed subsurface cavities are presented in Fig. 14 a,b. The lower-magnification image shows their distribution within the fracture surface (Fig. 14a), whereas the higher-magnification view reveals locally heterogeneous material and fragmented particles inside the cavities (Fig. 14b). Similar cavities had already been identified in the weld cross-section (Fig. 3c). Higher-magnification observations showed that they contained fragmented particles and locally heterogeneous material, most likely associated with the redistribution and mechanical breakup of pre-existing constituent particles during welding. Their exact nature, however, could not be determined solely from the fractographic observations.

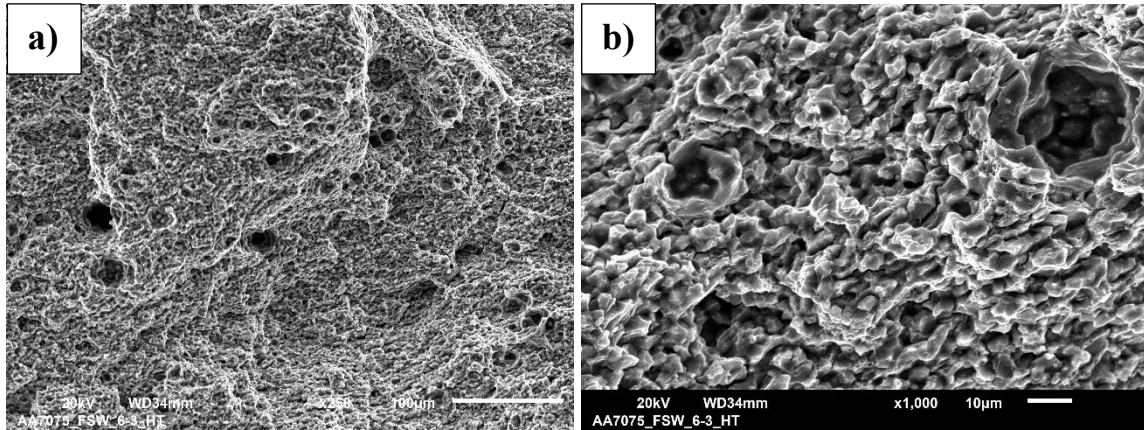


Fig. 14. Fracture surface of the 600+HT specimen showing (a) subsurface cavities and (b) their internal morphology at higher magnification.

The 600+HT specimen fractured within the SZ, slightly toward the retreating side, as shown previously in Fig. 13. In this condition, no AGG was observed in the PDZ, and the fracture therefore developed through the retained fine-grained structure. At lower magnification, the fracture surface exhibited a mixed morphology comprising relatively flat intergranular regions and rougher transgranular areas (Fig. 15a). Higher-magnification observations revealed fine dimples within the transgranular regions, confirming a local ductile contribution to the overall fracture process (Fig. 15b). This mixed morphology indicates the simultaneous operation of locally brittle and ductile fracture mechanisms despite the substantial increase in YS and UTS produced by post-weld heat treatment.

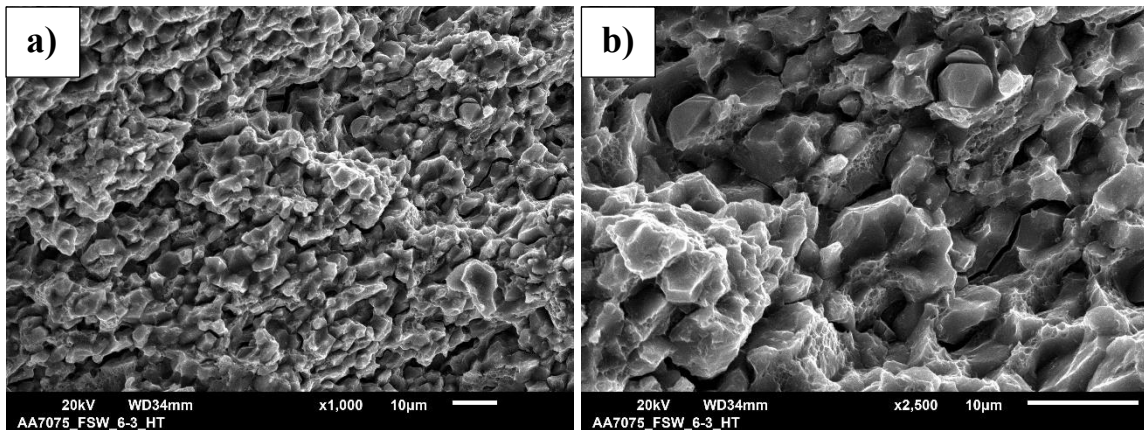


Fig. 15. Fracture surface of the 600+HT specimen showing (a) mixed intergranular and transgranular fracture morphology and (b) fine dimples within the transgranular regions.

For the specimens welded under water, fracture consistently occurred on the advancing side, near the boundary between the SZ and the adjacent TMAZ/HAZ region (Fig. 16a). In contrast, all specimens welded in quenching oil fractured within the HAZ on the retreating side (Fig. 16b). Thus, changing the cooling medium from water to quenching oil altered both the fracture location and the mechanical response of the joints, with the oil-cooled specimens exhibiting lower tensile strength. This indicates that the cooling conditions affected the local strength distribution across the joint and determined the region most susceptible to fracture.

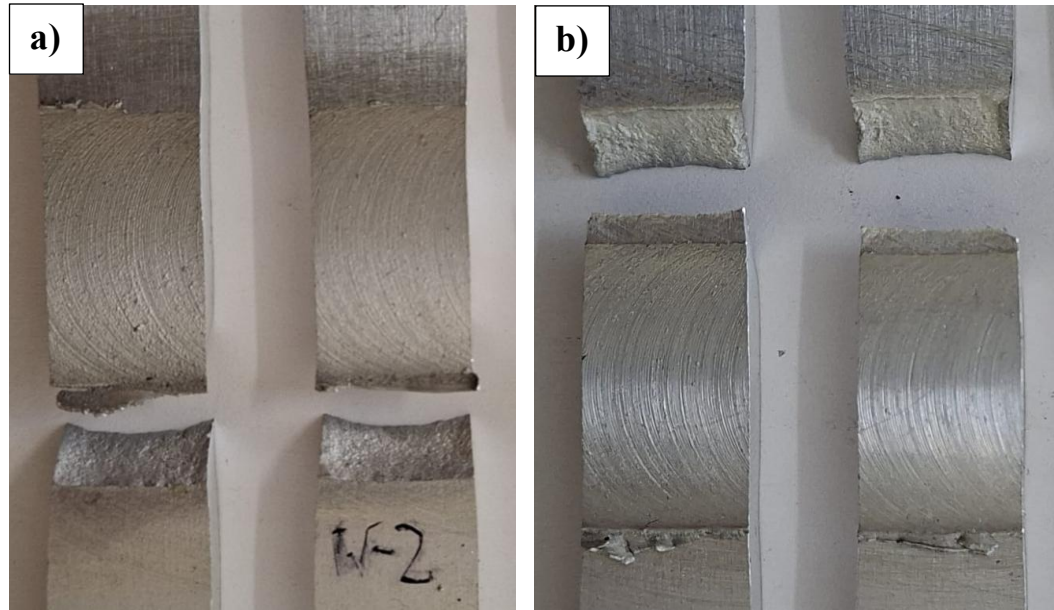


Fig. 16. Fracture locations after static tensile testing of specimens welded (a) under water and (b) in quenching oil.

The fracture surface of the specimen welded under water exhibited a more pronounced lamellar morphology, with numerous delaminations and elongated tearing features aligned with the rolling direction (Fig. 17a). These features reflect the anisotropic, rolling-induced structure of the sheet rather than a clear increase in macroscopic ductility, as the tensile-test results showed comparable elongation among the investigated specimens. In contrast, the specimen welded in quenching oil exhibited a less pronounced directional morphology and a greater number of particle-associated cavities and locally dimpled regions (Fig. 17b), indicating more frequent damage initiation around coarse second-phase particles.

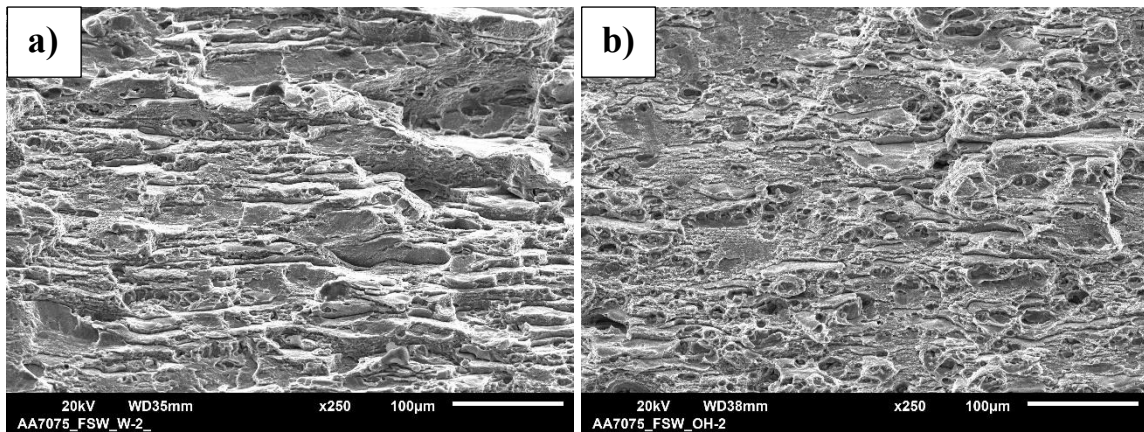


Fig. 17. Fracture surfaces of specimens welded (a) under water and (b) in quenching oil.

Post-weld heat treatment of the specimen welded under water resulted in a pronounced increase in strength. As shown in Fig. 18a, fracture occurred within the central region of the SZ, with the fracture path slightly shifted toward the advancing side. The fracture surface exhibited a highly heterogeneous morphology, in which individual layers formed by material flow during FSW could be clearly distinguished (Fig. 18b).

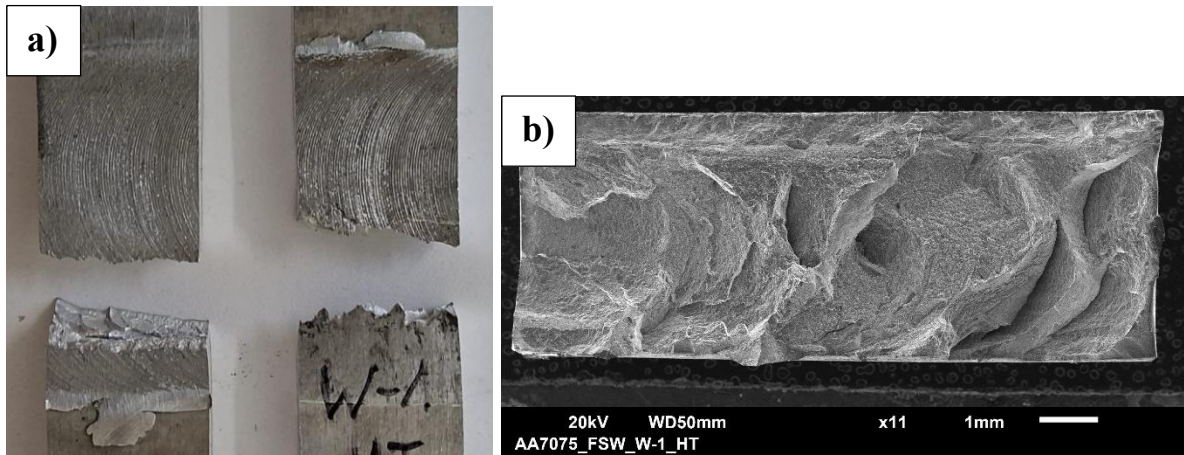


Fig. 18. Fracture of the Water+HT specimen: (a) fracture location after static tensile testing and (b) low-magnification view of the layered fracture-surface morphology.

The fracture morphology varied with the distance from the weld surface. At a depth of approximately 0.6 mm below the joint surface, a distinct layer about 0.2 mm thick was observed (Fig. 19a). This region exhibited a strongly refined morphology with numerous precipitate- or particle-associated cavities. Higher-magnification observations revealed predominantly dimpled features within this layer, indicating that ductile fracture was the prevailing local failure mechanism (Fig. 19b).

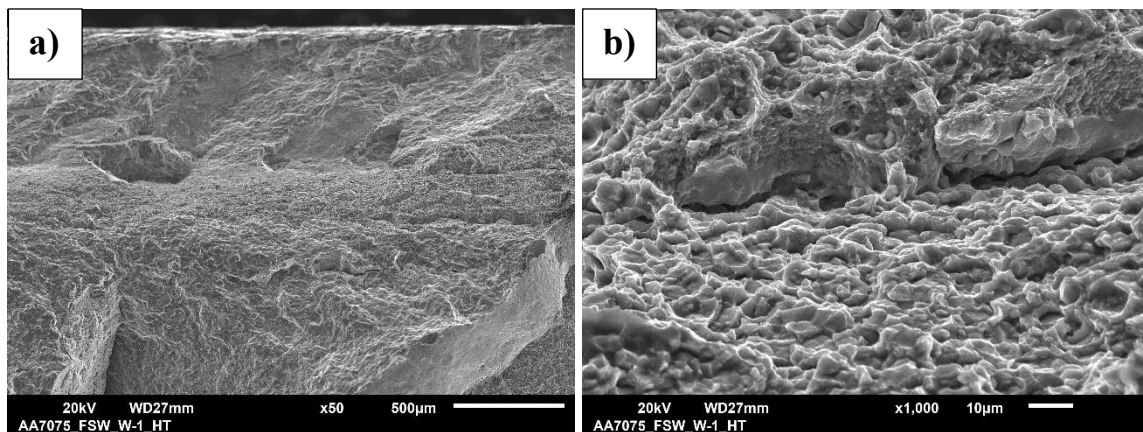


Fig. 19. Fracture surface of the Water+HT specimen showing (a) a distinct subsurface layer and (b) its dimpled morphology at higher magnification.

The marked differences in fracture morphology observed within the Water+HT specimen reflect the strong local heterogeneity of the SZ after PWHT. At a depth of approximately 0.8 mm below the joint surface, the fracture path entered the SZ, where the microstructure had been substantially modified by post-weld heat treatment. The fracture surface exhibited a coarse and heterogeneous morphology in the PDZ, consistent with the abnormal grain growth identified in this region during microstructural examination (Fig. 20a). Higher-magnification observations revealed irregular boundary features together with locally distributed cavities and second-phase particles, indicating the contribution of both intergranular cracking and localized ductile damage (Fig. 20b). In another region of the SZ, the fracture surface was dominated by a fine dimpled morphology, characteristic of microvoid nucleation, growth, and coalescence (Fig. 20c). At higher magnification, numerous coarse second-phase particles were observed within the dimples, with some of the larger particles showing evidence of fragmentation during fracture (Fig. 20d). These observations confirm that both locally brittle and ductile fracture mechanisms contributed to failure of the Water+HT specimen.

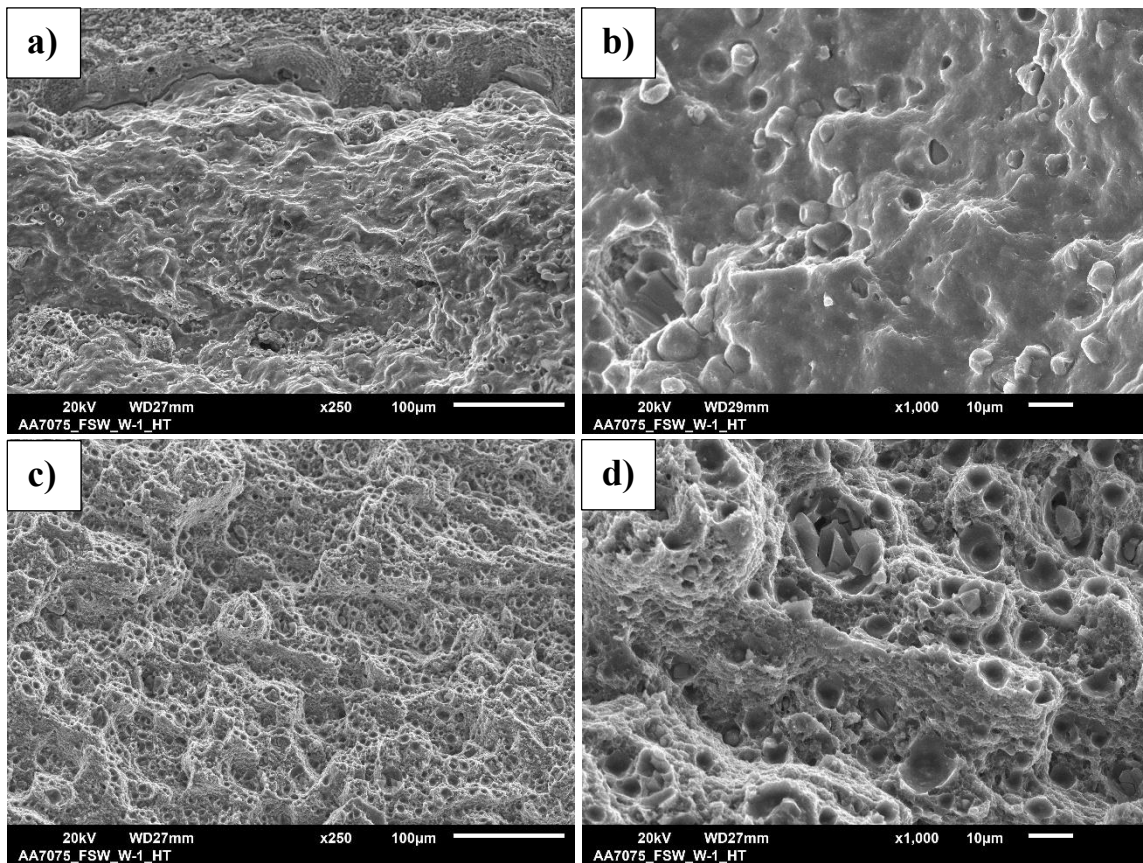


Fig. 20. Fracture surface of the Water+HT specimen within the SZ showing (a) coarse and heterogeneous morphology in the AGG-affected PDZ, (b) irregular boundary features with cavities and second-phase particles, (c) a predominantly dimpled region, and (d) coarse and locally fragmented particles within the dimples.

These observations confirm that both locally brittle and ductile fracture mechanisms contributed to failure of the Water+HT specimen. The pronounced local variation in fracture morphology is consistent with the observations of İpekoğlu et al. [20], who reported that PWHT of FSW AA7075 joints produced a heterogeneous SZ microstructure comprising AGG-affected

and retained fine-grained regions, with fracture preferentially developing along the interface between them. In the case of the specimens welded in quenching oil and subsequently heat-treated, fracture occurred within the weld, predominantly in its central region, with the fracture path slightly shifted toward the advancing side (Fig. 21a). In one specimen, partial pull-out of the SZ was observed, indicating a highly nonuniform fracture path. This behavior is consistent with the macrostructural observations, which revealed a substantial number of imperfections within the PDZ of the OH-70+HT joint. The corresponding fracture surface exhibited a strongly heterogeneous morphology, with distinct regions differing in roughness and local fracture mode (Fig. 21b).

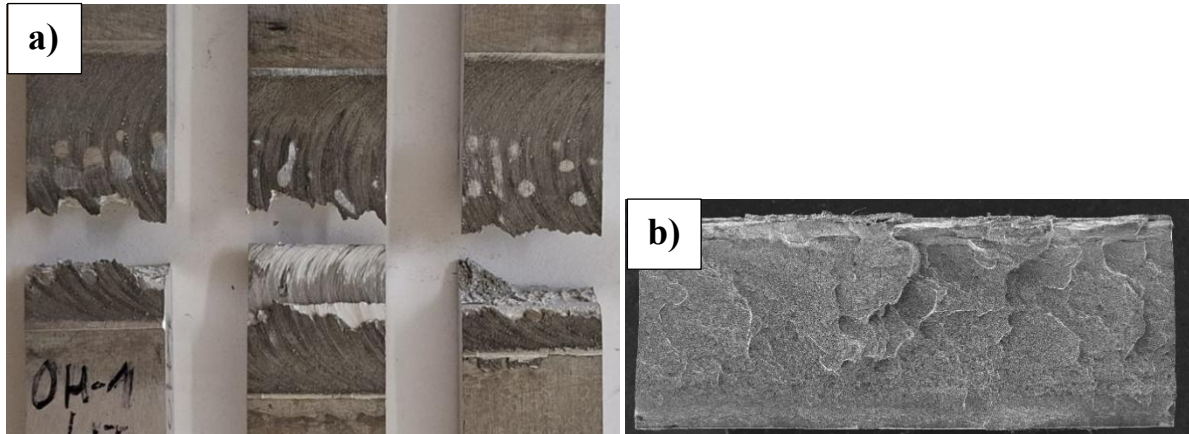


Fig. 21. Fracture behavior of the OH-70+HT specimens: (a) fracture locations and (b) heterogeneous fracture-surface morphology.

The fracture surface was dominated by a dimpled morphology, indicating that failure occurred primarily through microvoid nucleation, growth, and coalescence (Fig. 22a). Higher-magnification observations revealed larger cavities separated by plastically deformed material ligaments, confirming a locally ductile fracture mechanism in this region (Fig. 22b). The locally dense network of fine ligaments reflects extensive plastic deformation preceding final separation.

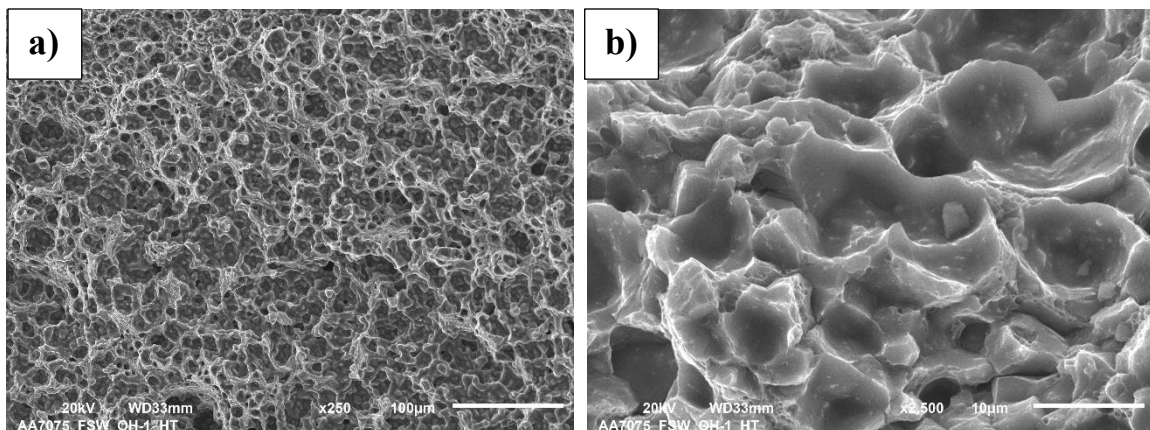


Fig. 22. Fracture surface of the OH-70+HT specimen showing (a) a predominantly dimpled morphology and (b) larger cavities separated by plastically deformed ligaments.

Although the fracture surface exhibited a predominantly ductile, dimpled morphology, the macroscopic elongation of the OH-70+HT specimen remained very low (Fig. 8a). This apparent discrepancy can be attributed to pronounced strain localization within the defect-rich SZ. Local stress concentration and increased stress triaxiality promoted rapid microvoid nucleation, growth, and coalescence within a narrow fracture zone, while only limited plastic deformation developed over the remaining gauge length.

4. Conclusions

The following conclusions can be drawn from this study:

1. Post-weld heat treatment increased and homogenized microhardness across all investigated joints, with the stir zone generally exhibiting higher hardness than the surrounding regions. This effect was attributed primarily to the precipitation-hardening response rather than to the retained fine-grained structure.
2. Abnormal grain growth occurred in the shoulder-driven zone of every heat-treated specimen, irrespective of the cooling condition or tool rotational speed. The pin-driven zone was also affected in most cases, demonstrating that neither accelerated cooling nor an increased rotational speed was sufficient to ensure microstructural stability during PWHT.
3. For defect-free joints, PWHT increased YS by more than 70% and UTS by approximately 35%, with the highest UTS values exceeding that of the base material. However, the mechanical response of defective joints remained governed primarily by welding- and heat-treatment-induced imperfections.
4. No straightforward relationship was identified between the extent of AGG and the tensile properties, including ductility. Fractographic observations revealed strongly heterogeneous failure mechanisms, comprising intergranular cracking, dimpled transgranular fracture, particle-assisted microvoid formation, and pronounced strain localization.

Author contributions Robert Kosturek developed the study concept, performed the friction stir welding experiments, conducted the microstructural characterization and microhardness measurements, analyzed the results, and prepared the manuscript. Krzysztof Grzelak carried out the post-weld heat treatment. Janusz Torzewski performed the static tensile tests. Tomasz Ślęzak conducted the fractographic analysis and prepared the corresponding section of the manuscript.

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Data and materials availability The datasets used and/or analyzed during this study are available upon reasonable request to the corresponding author.

Declarations

Ethical Approval and Consent to Participate The authors declare that all participants involved in this study provided informed consent to participate. Any data collected from participants was gathered in accordance with ethical standards and relevant guidelines.

Consent for publication The authors affirm that all co-authors have given consent for the publication of this manuscript.

Conflict of interest The authors declare that they have no conflicts of interest.

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