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Research Article

Effect of Grain Size on the Stress Corrosion Cracking of Ultrafine Grained Cu-10 wt% Zn Alloy in Ammonia

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The effect of grain size in the *micron* to submicron range on the stress corrosion cracking (SCC) of Cu-10 wt% Zn alloys was investigated using constant-load tests in ammonia vapor. The grain size was systematically varied from 4 μm to 0.12 μm by either cold-rolling or equal-channel angular pressing (ECAP), followed by annealing. The time to fracture increased with decreasing grain size above 1 μm but then began to decrease with decreasing grain size into the submicron range. This inverse trend in the submicron range is discussed in terms of a severe plastic deformation- (SPD-) induced ultrafine grain microstructure.

1. Introduction

While the effect of grain size reduction on the strength of metallic materials has been well established, its effect on corrosion is more complicated and does not appear to be explainable *using a universal law such as the Hall-Petch law of the yield stress*. It seems that the effect of grain size reduction on corrosion resistance is mostly positive in stainless steels [1] and aluminum alloys [2, 3], whereas the effect is marginal in copper [4, 5] and titanium alloys [6–8]. However, there are several contradictory reports involving the same materials and environment [9–11]. The limited available literature on the effect of grain size on stress corrosion cracking (SCC) [12, 13] reports an increasing resistance to SCC with decreasing grain size. Edmund investigated the effect of grain size on the SCC of α-brass in an ammonia environment using a constant-load test and reported increasing fracture time with decreasing grain size [14]. Additional discussion of the effect of grain size on corrosion can be found in comprehensive review papers [15, 16].

Severe plastic deformation (SPD) enables the reduction of grain size to the submicron range in bulk metallic materials for load-carrying structural applications [17]. The so-called ultrafine grained (UFG) materials formed by SPD exhibit unique physical and mechanical properties. For example, the strength increases enormously after ECAP and the ductility also remains relatively high or even increases in some cases [17–19]. These unique properties have frequently been attributed to deformation-induced UFG structures superimposed with the dislocation structure. The advent of SPD technology has rendered the corrosion behavior of UFG materials a more pressing issue and has brought the question of whether the dependence of corrosion properties on grain size can be extrapolated into the submicron range back to the scientific community [15].

The SCC susceptibility of UFG or nanostructured materials has been far less studied and much remains to be explored [20–31]. One limitation of SCC studies of SPD materials is that structural materials that exhibit SCC are generally high-strength alloys such as austenitic stainless steels and Cu-Zn alloys, which are difficult to process by SPD. Fortunately, corrosion studies only require a UFG structure in the surface layer, and surface modification of even very hard materials is possible using methods such as shot peening [32] or surface mechanical attrition treatment (SMAT) [33]. Another reason for the lack of research on this topic is the very large difference in yield strength between UFG materials formed by SPD and conventional coarse-grained materials, as a result of which the standard SCC tests that apply either a common load or displacement inevitably impose different levels of strain or

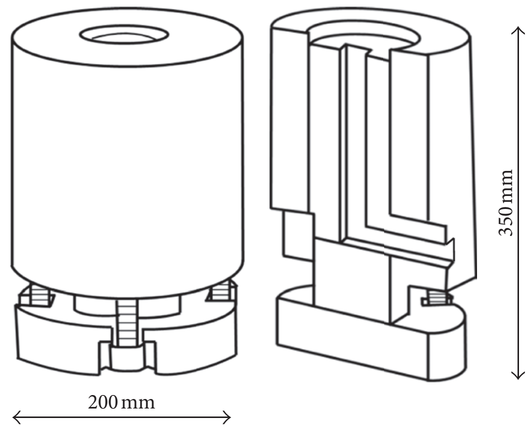

Figure 1: Schematic diagram and dimensions of the ECAP die.

Table 1: Processing recipes and grain size.

Sample	Deformation	Annealing	Grain size (μm)
1	None	None	38.0
2	CR 50%	673 K-60 min	12.6
3	CR 50%	623 K-60 min	3.30
4	ECAP 2 pass	623 K-15 min	1.10
5	ECAP 2 pass	623 K-10 min	0.71
6	ECAP 2 pass	623 K-5 min	0.44
7	ECAP 8 pass	473 K-10 min	0.15
8	ECAP 8 pass	473 K-1 min	0.12
9	ECAP 8 pass		0.12

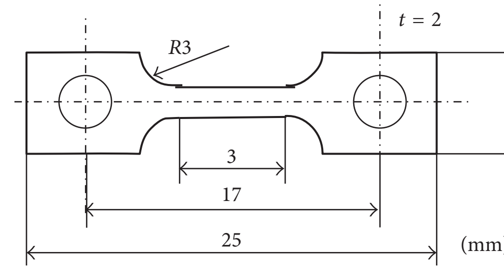

Figure 2: SCC specimen dimensions.

stress in these two materials. Therefore, these two approaches may yield considerable discrepancies in experimental results, for example, in the case of the time to fracture. As an alternative approach, these two very different materials have been compared by slow-strain-rate testing (SSRT), and their susceptibility to SCC was determined from the ratio of elongation to fracture in air to that in solution [20–22, 24, 26–29, 31]. However, it remains unclear whether the susceptibility to SCC determined by SSRT reflects performance in actual service environments. In this study, we investigated the effect of grain size, ranging from fine grains to submicron grains, on the susceptibility to SCC using a model Cu-10 wt% Zn-ammonia system under a common constant load. The objective of this study was not to compare the two extremes (UFG and conventional course-grained counterparts), as has been done in many previous studies, but to track the variation in SCC susceptibility in the transitional regime ranging from fine to submicron grains, to better understand the SCC of UFG materials from a physical point of view. As far as we know, this paper is the first to report the SCC susceptibility under constant-load tests for this range of grain sizes.

2. Materials and Methods

The grain sizes of commercial Cu-10 wt% Zn alloys were systematically controlled by cold-rolling and ECAP followed by heat treatment. For ECAP, 100 mm long billets with an 8 mm square cross-section were pressed through an ECAP die for either two or eight passes by the so-called Bc route, in which the sample is rotated by 90° around its longitudinal axis between passes. The ECAP die was deliberately designed so that harder materials such as brass and stainless steel can be pressed, as shown in Figure 1. Nevertheless, α-brass with a higher Zn content, for example, Cu-30 wt% Zn, is too hard for this ECAP die, even though it is more susceptible to SCC and would have been an appropriate material for the present study. Thus, the choice of Cu-10 wt% Zn was a compromise between the applicability to ECAP and susceptibility to SCC. Cold-rolling was carried out to a 50% reduction. *After ECAP and cold-rolling*, the samples were then annealed in an electronic furnace *as summarized in Table 1*, leading to grain sizes ranging from 0.12 μm to 38 μm. *Grain size was estimated using the intercept method, and twins were ignored following the rule of grain size measurement of copper and copper alloys described in JIS0501.*

The susceptibility to SCC was evaluated in constant-load tests using a cantilever-type apparatus (Figure 2) for specimens with a yield stress above 290 MPa. A common constant stress of 280 MPa was applied to all SCC specimens regardless of their yield stress and was selected based on our previous studies [25], which indicated that the susceptibility to SCC was much higher in as-ECAPed UFG Cu-10 wt% Zn than in the coarse-grained same alloys. The dog-bone specimens shown in Figure 3 were placed in a chamber in which a 14% ammoniacal solution was placed in the bottom to fill the chamber with ammonia vapor. The time to fracture was recorded by a stopwatch placed under the edge of the beam. The microstructure and fracture surface were examined by transmission electron microscopy (TEM, JSM 2100F) and field-emission-type scanning electron microscopy (FE-SEM, JSM-FE7001).

3. Results and Discussion

Nominal stress-strain curves obtained from tensile tests are shown in Figure 4. ECAPed materials had typical stress-strain curves with a high tensile strength and little strain hardening capability. Thus, they exhibited necking at the onset of plastic deformation. The tensile stress exceeded 600 MPa, much greater than that of pure copper (approximately 450 MPa

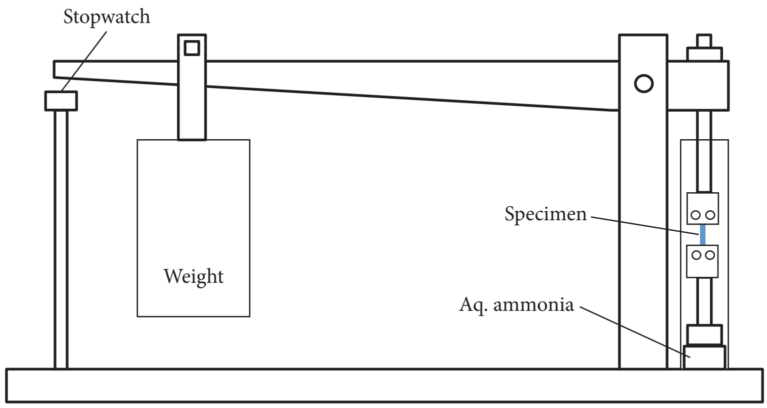

Figure 3: Schematic diagram of the constant-load SCC test.

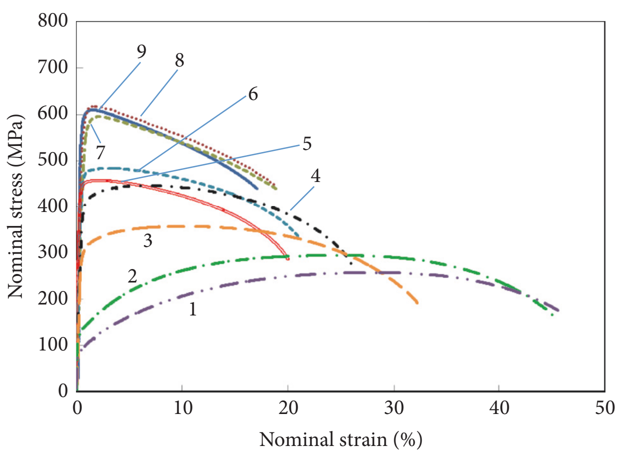

Figure 4: Nominal stress-strain curves of tensile tests. Numbers 1–9 correspond to the sample numbers in Table 1.

after 8 passes of ECAP). This higher tensile stress was mainly caused by solid-solution hardening and a lower stacking fault energy (SFE). Cu-10 wt% Zn has an SFE of 35 mJ/m^2, which is much lower than that of pure Cu (78 mJ/m^2) [34]. Microstructures observed by TEM and SEM are shown in Figure 5, and the processing routes and resulting grain sizes are summarized in Table 1. The minimum grain size of 0.12 μm was achieved after 8 passes of ECAP and is somewhat smaller than that achieved with pure copper [5, 31] and pure aluminum [35] in our previous studies. This smaller grain size achieved by SPD is associated with a lower SFE, and several studies have demonstrated that the minimum grain size achieved by SPD scales with the SFE [36–39]. The larger grain size shown in Figure 5 was obtained by recrystallization and/or grain growth using appropriate combinations of ECA/cold-rolling and annealing. Whereas a relatively uniform grain size was observed in samples processed by ECAP, samples processed by cold-rolling were less uniform, indicating that continuous grain growth occurred in the former case while discontinuous recrystallization occurred in the latter. Several studies have reported that UFG structures with a high fraction of high-angle grain boundaries (HAGBs) exhibit continuous grain growth during annealing after a very high strain is imposed by SPD, whereas discontinuous grain growth or recrystallization tends to occur when the HAGB fraction is lower and less strain is imposed by SPD [40–44]. The relationship between yield stress and the reciprocal root of grain size is shown in Figure 6. Hall-Petch relationship can be divided into two regions with different slopes: Region I with grain sizes larger than 1 μm and Region II with smaller grain sizes. The slope corresponds to the constant k in the Hall-Petch relationship, represented as $\sigma_y = \sigma_o + k/\sqrt{d}$, where σ_y is the yield stress, σ_o is a constant, and d is the grain size. The constant k was estimated to be 0.40 MPam$^{1/2}$, which is not far from 0.27 MPam$^{1/2}$ reported by Armstrong et al. [45]. A transition of Hall-Petch slope with decreasing grain size in the submicron range was reported for UFG aluminum processed by ARB [46]. The lower slope for grain sizes smaller than 1 μm was estimated to be 0.07 and attributed to residual mobile dislocations inside the grains that carry plastic strain. For larger grain sizes after longer annealing times, the slope becomes steeper because of reduced density of mobile dislocations inside the grains [46]. This discussion was based on the assumption that high-angle grain boundaries act as sinks of mobile dislocations, consuming them during post-SPD annealing [47]. Similarly, in Region II in our studies, some residual mobile dislocations may carry plastic strain. However, in comparison with pure copper and pure aluminum with a higher SFE, dislocations are extended into Shockley partial dislocations with stacking faults and are difficult to be absorbed into grain boundaries, as discussed later.

In the SCC tests, the specimens were covered with a black tarnish film, which is well known to be a thick brittle copper oxide (Cu_2O) that causes intergranular SCC under tensile stress [48–50]. In Cu-Zn alloys with less than 20 wt% Zn, intergranular SCC tends to occur, whereas alloys with more than 20 wt% Zn tend to exhibit transgranular SCC [51, 52]. Cracks propagated in the tarnish film and formed at a higher rate on grain boundaries than within grains. Residual dislocations along the grain boundaries may enhance the

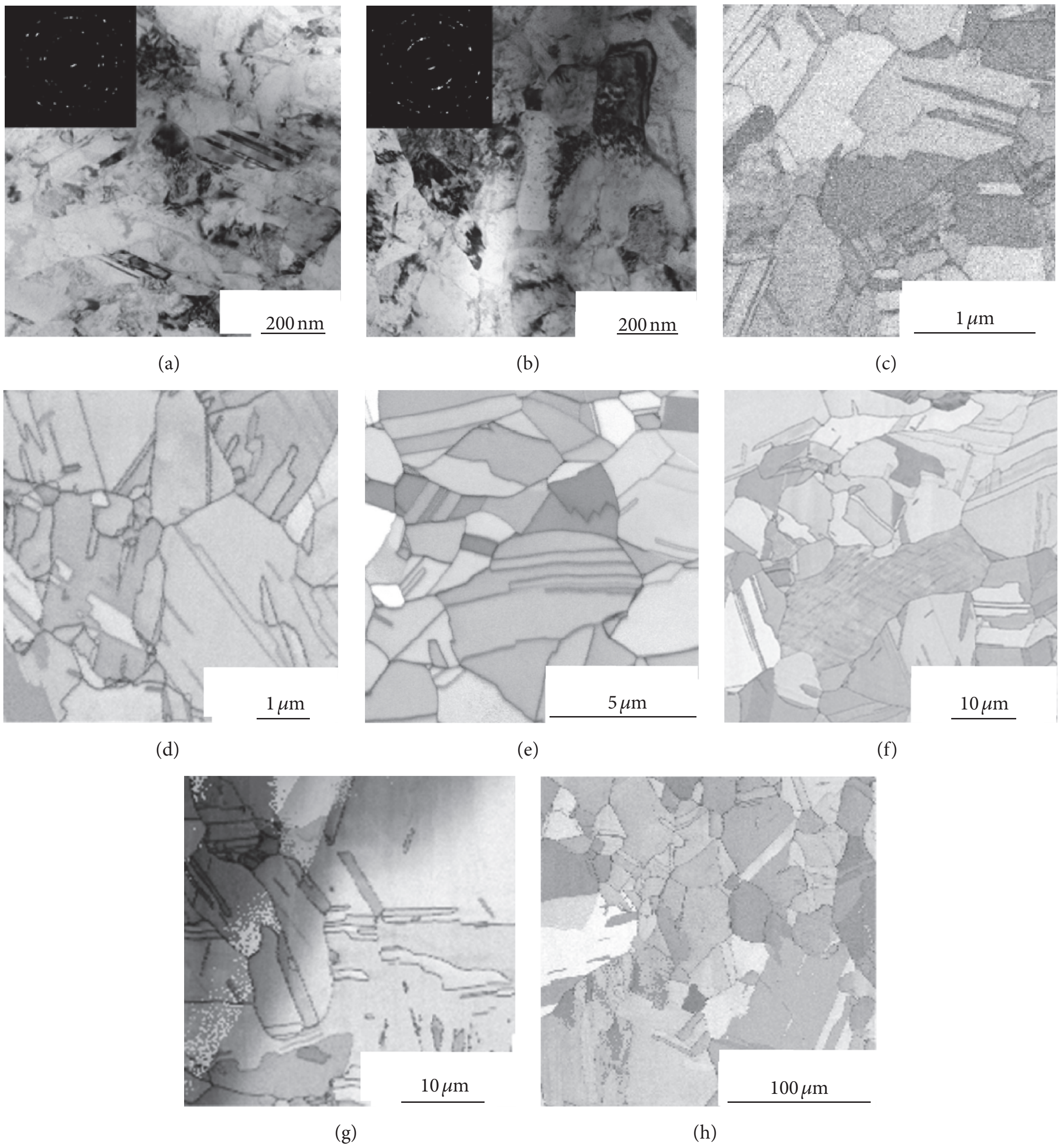

Figure 5: Microstructure after ECAP/cold-rolling and final annealing of (a) sample 8, (b) sample 7, (c) sample 6, (d) sample 5, (e) sample 4, (f) sample 3, (g) sample 2, and (h) sample 1.

reactivity of the grain boundaries with ammonia and the susceptibility to IGSCC. Figure 7 shows times to fracture obtained in SCC tests as a function of the inverse square root of grain size. Data for Cu-30 wt% Zn in ammonia from Edmund [14] is also plotted in the figure. As with the yield stress shown in Figure 6, the variation can be divided into two regimes, with a border at 1 μm. Considering Edmund's data for Cu-30 wt% Zn with large grains, it seems reasonable that the time to fracture increases with decreasing grain size from the conventional coarse grain regime down to 1 μm (Region I) but then decreases with decreasing grain size in Region II. The positive trend in Region I can be explained on the basis of the assumption that an intergranular crack initiates and propagates when local stress concentration by dislocation accumulation at the grain boundary reaches a certain critical value, so at smaller grain sizes, a higher applied stress is required to activate enough dislocations [53]. The changes in SCC susceptibility for grain sizes smaller than 1 μm (Region II) may be closely associated with the change in the Hall-Petch slope and can be attributed to residual dislocations, mobile or immobile, in the grains. These dislocations reside along grain boundaries or are trapped at the grain boundaries in a nonequilibrium state [54] and enhance the reactivity of grain boundaries in a corrosive environment in Region II, rendering the susceptibility less dependent on grain size. In our previous studies of UFG pure copper,

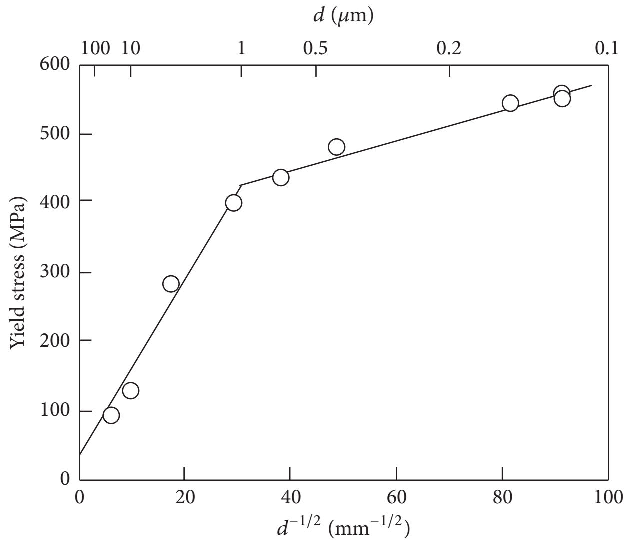

FIGURE 6: Relationship between yield stress and the inverse square root of grain size.

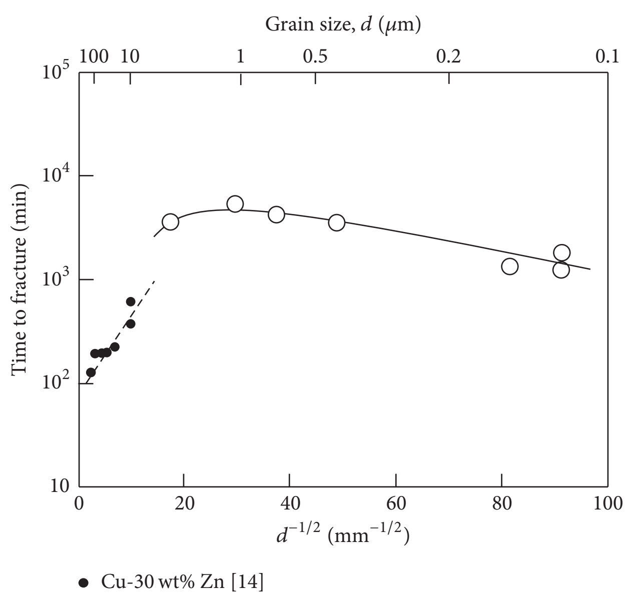

FIGURE 7: Relationship between time to fracture and the inverse square root of grain size in the SCC tests.

very short annealing for 90 seconds at 200°C relaxed the nonequilibrium grain boundaries to their equilibrium state with little grain growth [5, 31, 55, 56]. However, the extended dislocations with stacking faults in Cu-Zn alloys are expected to be difficult to be absorbed into the grain boundaries as a result of the lower stacking energy and therefore may remain in the grains during annealing for longer times at higher temperatures. Furthermore, the nonequilibrium grain boundaries may be more stable due to Zn segregation, and some may remain in Region II [57–59]. A common constant stress of 280 MPa was applied to all specimens, so the applied stress normalized by yield stress, σ_a/σ_{ys}, was lower in specimens with a smaller grain size, which have a higher yield strength. This means that even though the dislocation activity under macroscopic elastic deformation is lower in these materials, they are more susceptible to SCC.

Grain boundary sliding is considered to be one possible mode of plastic deformation in UFG materials [18, 60] and has been observed at room temperature in UFG pure aluminum by atomic force microscopy [61]. Film rupture facilitated by grain boundary sliding at the crack tip may enhance the susceptibility to IGSCC under a constant load. The present results are compatible with our previous studies that employed compact specimens of Cu-10 wt% Zn, in which the threshold stress was lower and the time to fracture was higher in UFG samples than in their counterparts with larger grain sizes [25].

Macroscopically, all of the SCC specimens fractured in a brittle manner with little plastic deformation. SEM fractographs of coarse-grained sample 1 and UFG sample 9 are shown in Figures 8(a) and 8(b). Microscopic observation at higher magnification revealed an intergranular mode of fracture in both samples (Figures 8(c) and 8(d)). Since the grain size was very small and the fracture surface was readily covered by a tarnish film, it was difficult to obtain clear evidence of IGSCC by SEM observation.

In addition to grain size, grain boundary structure is another important factor influencing IGSCC resistance [62–64], and several attempts have been made to alleviate SCC by controlling the grain boundary character distribution (GBCD) or by an alternative approach called grain boundary engineering (GBE) [65–68]. Ideally, the grain size and grain boundary character distribution should both be considered when characterizing the SCC of UFG materials by SPD. Therefore, SPD still has potential for high SCC resistance materials through the equilibration and control of grain boundary character distribution.

4. Conclusions

In this work, the effect of decreasing grain size into the submicron regime on the SCC of Cu-10 wt% Zn alloys in ammonia was investigated using a constant-load test, leading to the following conclusions:

(1) The yield stress obtained by tensile tests increased with decreasing grain size, but the tendency decreased for grain sizes smaller than 1 μm. Thus, the Hall-Petch relationship can be divided into two regions with different slopes, with a lower slope for grain sizes smaller than 1 μm.

(2) The time to fracture by SCC increased with decreasing grain size down to 1 μm but then decreased with further decreases in grain size into the submicron scale. In other words, there was a critical grain size above which the susceptibility began to increase, and this grain size matched the grain size that divides the two Hall-Petch relationships.

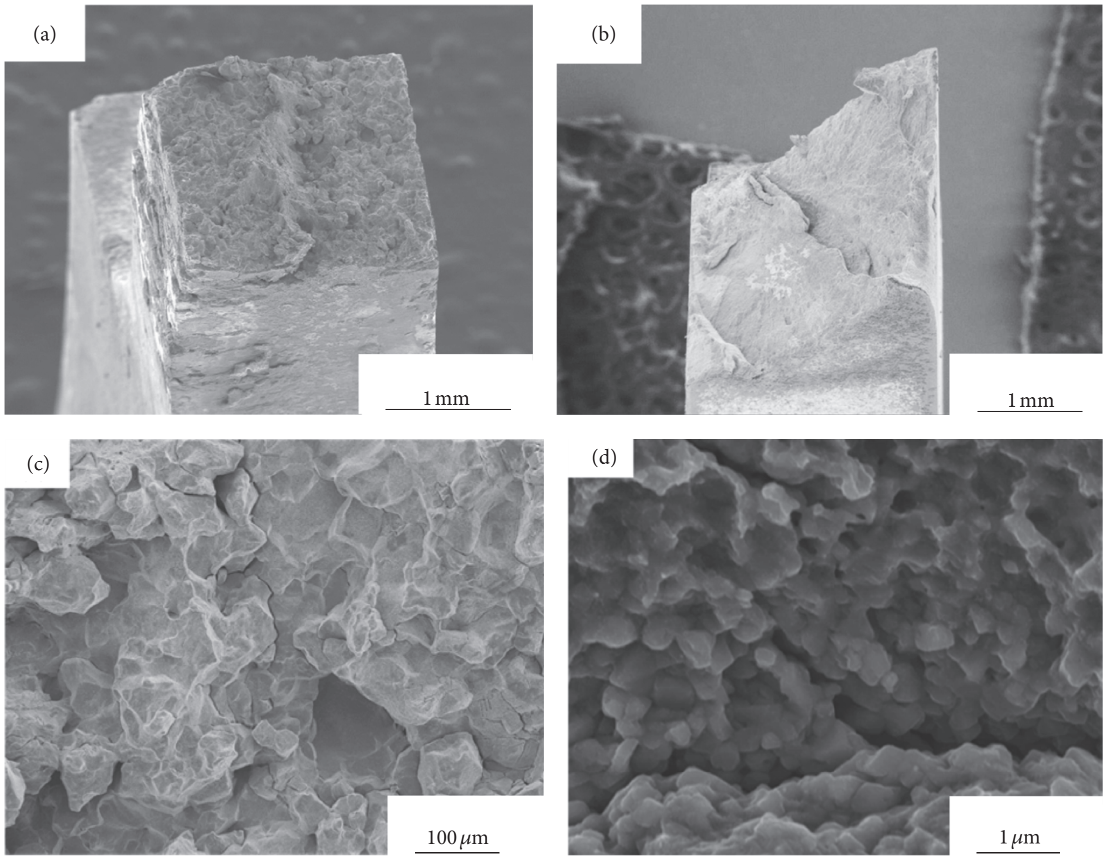

Figure 8: Fracture surfaces after SCC tests of (a), (c) sample 1, and (b), (d) sample 9 in (a), (b) macroscopic appearance, and (c), (d) microscopic observation.

(3) Stress corrosion cracks propagated intergranularly regardless of grain size. SPD-induced grain boundaries have a high sensitivity to chemical reaction and intergranular SCC.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

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