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Case study

Metallurgical investigation of cracked Al–5.5Zn–2.5Mg–1.5Cu aluminium alloy valve

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ABSTRACT

The high strength aluminium alloy Al–5.5Zn–2.5Mg–1.5Cu (AA7075) is being widely used in realisation of aerospace components. A component 'fill and vent valve' used in liquid propulsion system was fabricated from AA 7075 forgings in T7352 temper condition, and subsequently undergone various functional tests, four years back. Recently, during dye penetrant test after proof pressure test at 525 bar, a valve indicated presence of a crack. Detailed metallurgical investigation indicated that failure was caused by stress corrosion cracking.

1. Introduction

Aluminium alloy Al–5.5Zn–2.5Mg–1.5Cu (AA 7075) belongs to high strength category and is being extensively used for a number of non-weldable components in space programme. One such component namely fill and vent valve was fabricated out of AA 7075 forged block in T7352 temper condition through machining. These components in anodised condition were subjected to various functional tests before acceptance and thereafter stored in bonded store for a period of five years. Recently, one of the valve was proof pressure tested (PPT) at 525 bar. Subsequent to the PPT, routine dye–penetrant test revealed presence of cracks (Fig. 1). Detailed metallurgical investigation was carried out to understand the cause of failure. This paper brings out the detail of investigation and recommendation thereafter.

2. Material

The valve was fabricated from AA 7075-T7352 cylindrical forged block of size 75 mm diameter × 250 mm length. Cut pieces from the 116 mm diameter extruded billet were forged through successive steps of upsetting and drawing to realise the required forged block. Forged blocks were heat treated to T7352 temper condition, which calls for solutionising at 475 °C for 4 h, water quenching, cold compression 2–3% followed by two step ageing treatment (107 °C– 6 h–AC + 177 °C-7 h–AC).

Mechanical properties for all such forged blocks duly tempered to T7352 condition were UTS: 440–507 MPa, 0.2% PS: 356–458 MPa, %E: 8.3–12.7 and electrical conductivity: 38–40.5% IACS. The chemical composition of the failed component was evaluated and was confirmed to be AA 7075 (Table 1).

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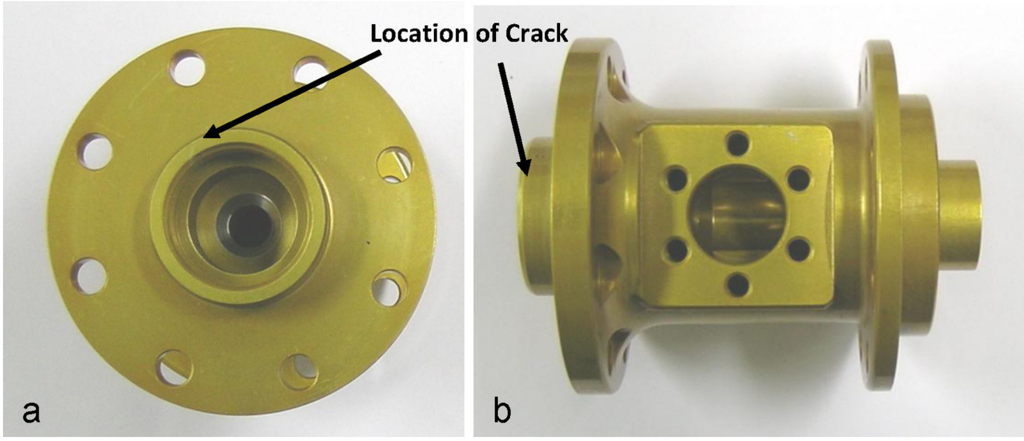

Fig. 1. Photograph showing the component (a) radial plane and (b) axial plane.

Table 1
Chemical composition of cracked valve.

Zn	Mg	Cu	Cr	Ti	Si	Mn	Al
5.8	2.41	1.68	0.24	0.02	0.16	0.11	Rest

3. Observation

The crack was seen on a radial plane of component, coincided with short transverse plane of the forged stock used to realise the component. Crack propagation was confirmed to its axial plane. The cracked valve was observed under optical microscope with stereographic facility. Numerous corrosion pits were seen on radial as well as axial plane at the vicinity of the crack (Fig. 2).

The cut pieces from the cracked valve were removed with utmost care and subjected to metallurgical investigation using optical and scanning electron microscopy to elucidate the cause of failure. All along the crack traverse at many locations on radial as well as axial plane, spalling of anodised layer was noticed (Fig. 3). Feature of corrosion underneath of loosely held anodised layer was also noticed as shown by arrow (Fig. 3a).

It was understood during the investigation that there were reasonable delay between final machining of component and their anodising.

Pits nearby cracked region and the corrosion product within the cavity was seen (Fig. 4) on radial plane of component. There were few more additional locations, where corrosion initiated and resulted in minor pits, encircled with dotted line (Fig. 4). Similar was the observation at crack traverse on axial plane (Fig. 5). The axial plane of cut pieces removed from failed valve and consisted of main crack was polished using conventional metallurgical techniques. The polished specimen was viewed under SEM to study crack tip morphology. Few fine additional cracks running parallel to the primary crack was noticed (Fig. 6). At some locations such fine cracks had shown crack branching. The crack branching was confined to grain

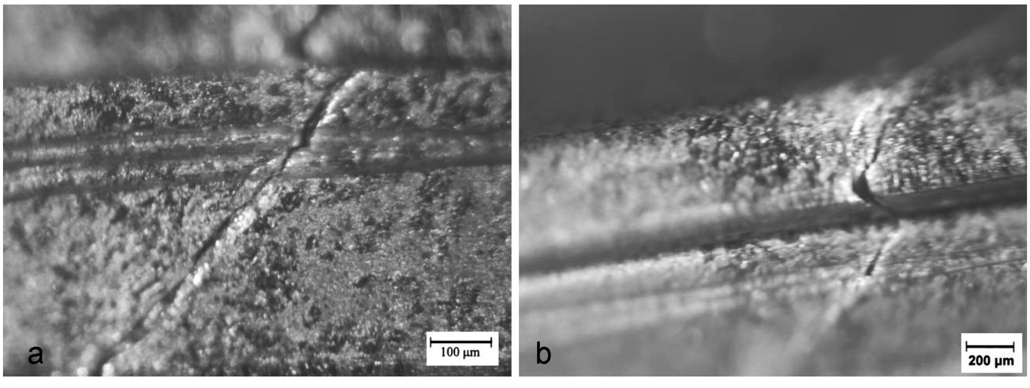

Fig. 2. Stereo photomicrographs showing cracks on (a) radial and (b) axial plane with numerous corrosion pits surrounding the crack.

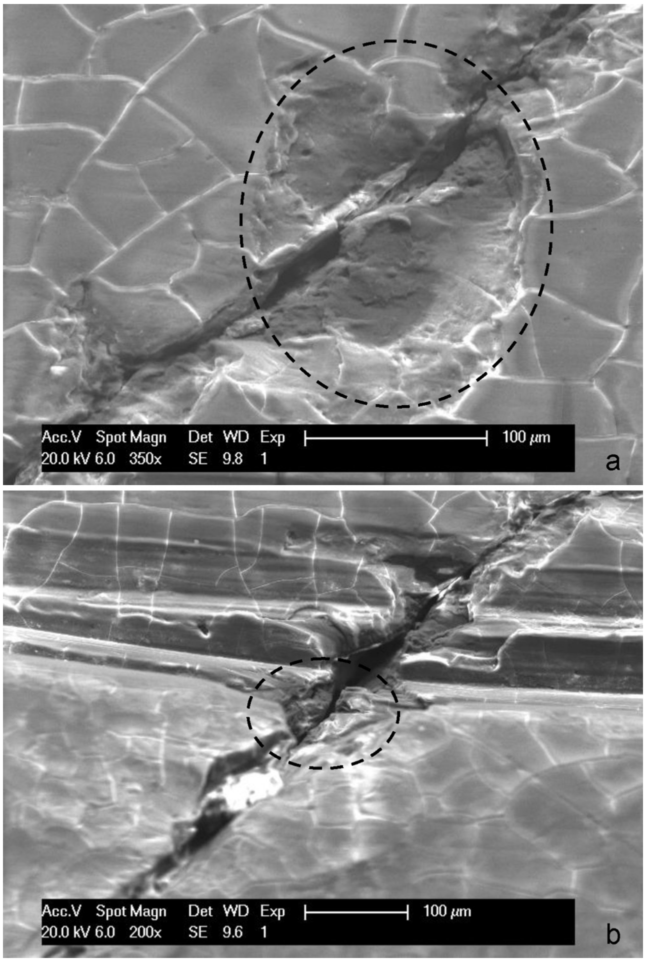

Fig. 3. SEM photomicrographs showing (a) crack on radial plane and devoid of anodized layer, (b) crack on axial plane.

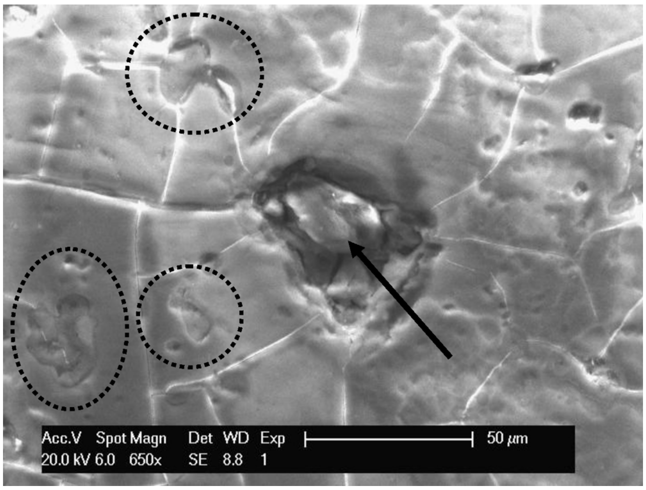

Fig. 4. SEM photomicrograph showing the corrosion pits (shown by arrow) adjacent to the crack on radial plane and locations of corrosion initiation (dotted encircled).

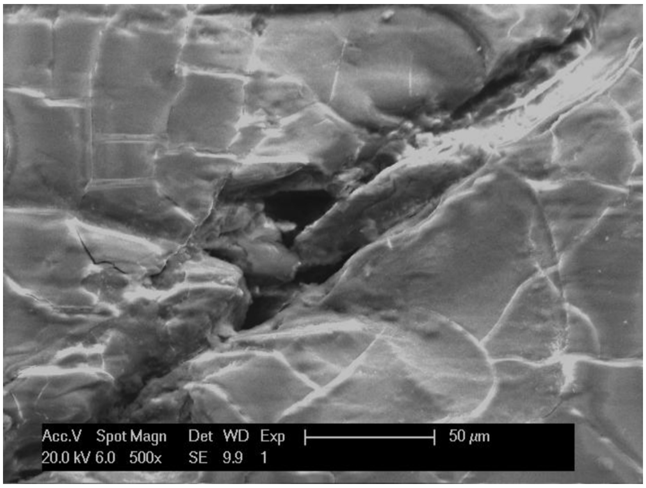

Fig. 5. SEM photomicrograph showing crack on radial plane with feature of grain dissolution.

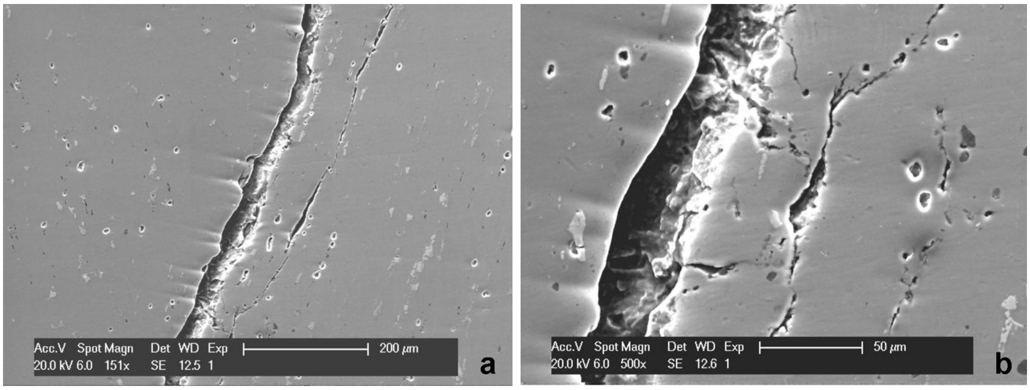

Fig. 6. SEM photomicrographs showing (a and b) multiple cracks running parallel to primary crack.

boundaries of recrystallised grains. There were spots (isolated) of preferential anodic dissolution sites. Crack tip was sharp and resembled features of as seen for traverse direction along grain boundaries (Fig. 7). The polished specimen, duly etched with Keller's reagent [2 ml HF + 3 ml HCl + 5 ml HNO_3 + 190 ml H_2O] revealed crack propagation along grain boundaries. Features of crack branching were also seen (Figs. 8 and 9). Traverse of crack tip all along the grain boundaries was confirmed as shown in Fig. 9b.

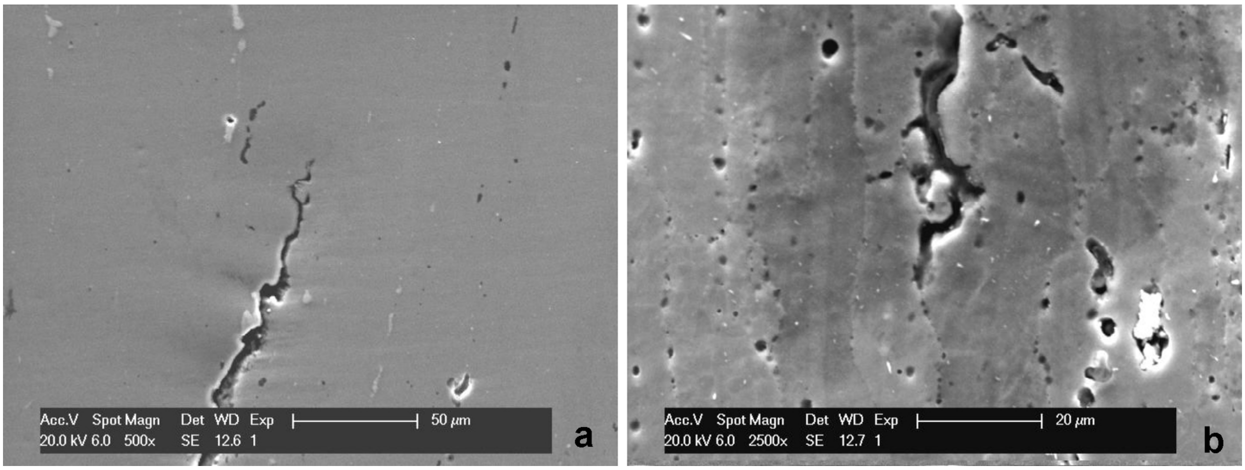

Fig. 7. SEM photomicrograph showing crack tip, confirming dissolution product within crack opening.

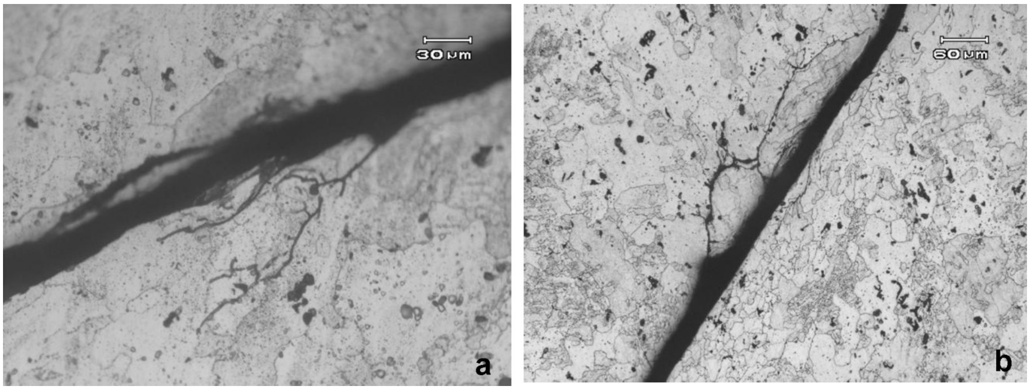

Fig. 8. Optical photomicrographs showing (a and b) crack branching along grain boundaries.

The fracture surface was opened carefully and observed under SEM. Fracture surface was fully covered with corrosion product, however presence of secondary cracks running parallel to each other was seen (Fig. 10) and was consistent with boundaries of elongated grains. Microstructure of region away from cracked region was seen on radial and axial plane. Longitudinal plane had elongated grains with grain aspect ratio of 12 and above. At many locations dried mud pattern of corrosion product (Fig. 10), typical of SCC [1] was seen at fracture surface. Electrical conductivity measurements were also carried out on the samples taken from the cracked component to ascertain whether the compression after solution treatment

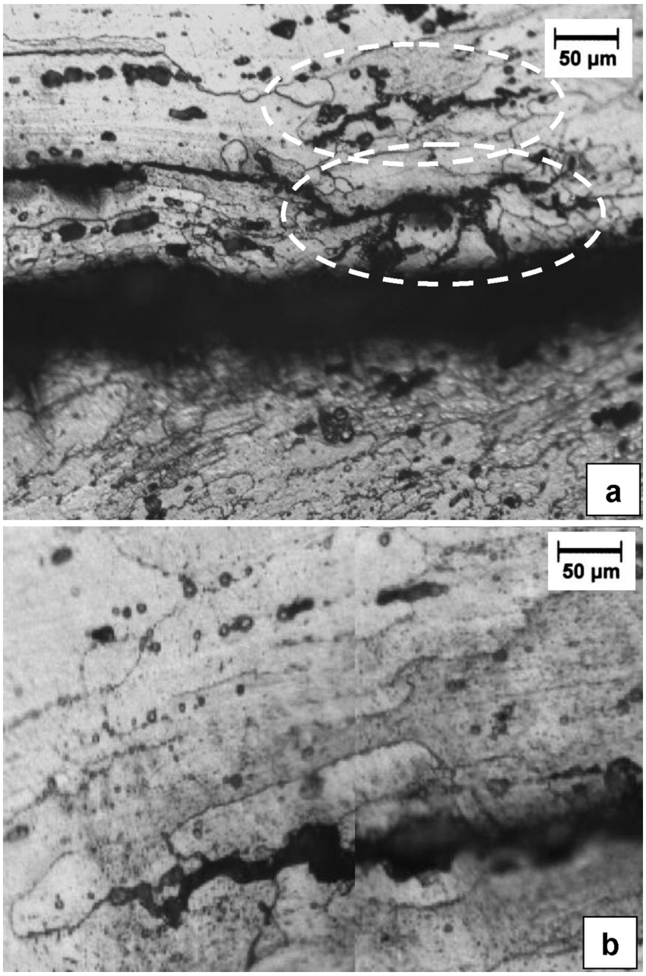

Fig. 9. Optical photomicrographs showing (a) multiple cracks running along parallel to primary crack and (b) crack tip traverse along grain boundaries.

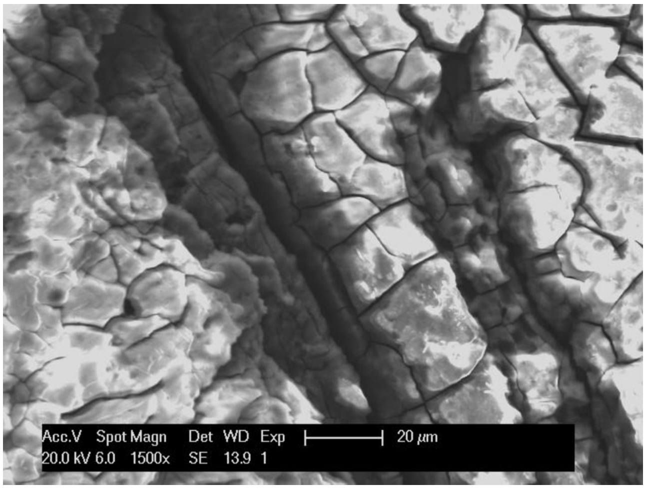

Fig. 10. SEM fractograph showing dried mud pattern of corrosion product on fracture surface.

and prior to ageing and/or the ageing temperature/time was sufficient to meet the specified minimum value of electrical conductivity (40% IACS) for T7352 treatment [2]. The measured conductivity on specimen taken out from the failed component was 39% IACS.

4. Discussion

Cracking occurred on radial plane of component, which was short transverse plane of forged block used to realise the component. Crack penetrated in axial plane with its path along the grain boundaries and had its branching at many locations, leaving behind the dried mud pattern of the corrosion product on the fracture surface. Such evidences were confirmative for the cause as stress corrosion cracking (SCC).

The influence of microstructure on susceptibility of Al–Zn–Mg–Cu alloys to SCC has been reported to strongly depend on their chemical composition and heat treatment condition. The composition, size and distribution of grain boundary phase/ precipitates are the determining factors to the susceptibility of Al–Zn–Mg–Cu alloy. AA 7075 alloy is known to be highly susceptible to stress corrosion cracking when used in chloride environment and in peak aged condition, especially in short transverse direction [3]. The classical solution to this problem is to bring the alloy to a condition of two-stage ageing [4,5], which exhibits good resistance to SCC. The prolonged ageing of Al–Zn–Mg–Cu resulted in decrease in susceptibility to SCC. This is due to the change in volume and distribution of strengthening $MgZn_2$ phase precipitates [6].

The temper condition T7352, which employ 2–4% cold compression after solution treatment and prior to two-step ageing treatment, provides better SCC resistance by reducing the locked-in stresses and modifying the shape and type of precipitates. In the present case study, forged stock were given compression in the range of 2.31–3.5%. The microstructure difference amongst the T6 and T7352 temper of Cu bearing Al–Zn alloy (AA 7075) differ in size and type of precipitates, which changes from predominantly Gunnier–Preston (GP) zone in T6 temper to ή, the metastable transition form of η ($MgZn_2$) in T7352. None of these differences can be detected by optical metallography. Even the resolution possible in electron microscope is insufficient for determining whether the precipitation reaction has been adequate to ensure the expected level of resistance to SCC [7]. However, for quality assurance, Cu containing 7XXX alloy in T73 or T7352 tempers are required to have specified minimum value of electrical conductivity (40% IACS).

In the present case, the measured conductivity values on the forged stock of lot were in the range of 38.0–40.5% IACS. It is understood that the forged stock was solution treated at 478 °C-6 h-WQ, cold compressed (2.3–3.5%) and aged (two stage) at 107 °C -8 h-AC + 177 °C–8 h-AC. The achieved conductivity was in the range of 39.7 to 40.4%IACS, however YS: 328– 363 MPa and UTS: 400–428 MPa were lower. The forged stock was re-solutionised with little modification in two steps ageing, i.e. 107 °C-6 h-AC + 177 °C -7 h–AC. The forged stock, duly aged (modified) condition has reasonably good strength. However out of 3 specimen taken from longitudinal plane of forging had high PS (475 MPa) than that of other two values (380 and 423 MPa). The specimen with high PS value had conductivity in the range of 38.1–38.7% IACS.

The conductivity value of more than 40% IACS is most desirable criterion for material acceptance in T7352 condition. However, in case if conductivity lie in the range of 38–40% IACS and 0.2% PS of material does not exceed 82 MPa more than the minimum specified 0.2% PS value, the lot can be accepted.

In the present case, since the one value of 0.2%PS (457 MPa) is more than 0.2%PS (min) + 82 MPa, additional test (twice the number) was carried out, which were within the minimum 0.2%PS + 82 MPa limit.

Further time gap between the machining and anodising was found to be reasonably high. Keeping machined component unprotected for such longer duration of 11–14 months is not desirable, as it may have deleterious effect and cause corrosion.

The forging stock undergone re-solutionising treatment and had marginally higher PS along with conductivity on lower side within the band of 38–40% IACS.

The another important controlling factor, effecting resistance of a particular wrought alloy to SCC is the direction of stressing with respect to elongated grain structure. Maximum susceptibility occur if stressing is normal to the grain direction i.e. in the short transverse direction of components, especially when grain aspect ratio is high. This is because the crack path along the grain boundaries is so clearly defined [8]. The stress corrosion cracking propagated very rapidly parallel to the rolling direction [9]. In the present case, grain aspect ratio of the value 12 and above defined the grain boundaries of the elongated grains in direction perpendicular to the valve radial face and thus facilitated the crack propagation.

The component was anodised more than five years ago and after qualification kept in store. The presence of numerous pits on the valve face, especially nearby cracked region was indicative of anodised layer inability to prevent corrosion. The anodised layer was not intact throughout. There were many locations all along the crack, indicating devoid of anodised layer. It seems as if the anodised layer was mechanically damaged/or deteriorated due to long storage period, which facilitated corrosion to take place. In fact, under such condition the anode to cathode area ratio will be drastically low, resulting in very high anodic current density and in order to balance during the electro chemical reactions, the corrosion will be severe. Once anodised layer is broken, and corrosion initiated with high current density, its penetration through anodic Cu-depleted zones surrounding the precipitates at grain boundaries goes uncontrolled. Evidence of anodic dissolution at crack tip (Figs. 7b and 9b) and presence of dried mud pattern of corrosion product on fracture surface support this conclusion. Further, resistance of this alloy to SCC is inferior, when conductivity is on lower side of the band 38–40% IACS, that too with 0.2% PS on higher side. It is always preferred to use AA 7075-T7352 material with conductivity more than 40% IACS. In the present case, little modification in ageing temperature/time may yield conductivity more than 40% IACS with strength marginally above minimum required value. Further the time gap between final machining and anodising must be kept as low as possible.

5. Conclusion

The valve failure occurred due to stress corrosion cracking.

Recommendations

1. It is desirable to use material in T7352 condition with conductivity more than 40% IACS. The ageing cycles may be modified accordingly.
2. Time gap between the machining and anodising should be kept bare minimum.
3. Components, if stored for longer period, adequacy of anodised layer should be ensured.

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