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Microstructural processes occurring during creep of friction stir welded AA2024-T3 alloy

Mikrostrukturni procesi koji se odvijaju tokom puzanja legure AA2024- T3 zavarene trenjem sa mešanjem

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Abstract

The poor weldability of AA2024 aluminum alloy limits its use for industrial applications. Being a non-fusion welding process, Friction Stir Welding (FSW) seems to be a promising solution for welding this alloy. FSW was applied in the current study in order to butt weld AA2024-T3 aluminum alloy plates and to study the creep behavior of the weld. Creep tests were conducted at 250 °C and 315 °C both on the parent material and on the friction stir welded specimens. A comprehensive Transmission Electron Microscopy (TEM) study together with High Resolution Scanning Electron Microscopy (HRSEM) study and Energy Dispersive X-ray Spectroscopy (EDS) analysis were conducted in order to investigate the microstructural processes. The parent material seems to contain two kinds of Cu-rich precipitates - coarse precipitates having the size of a few microns each and uniformly dispersed fine nanosized precipitates. However, this microstructure was found to be unstable at the temperature range of 250-315 °C, secondary precipitation was found to take place, this secondary precipitation is responsible for grain boundary decoration and the appearance of secondary rod-shaped precipitates and for some degree of coarsening of the nanosized precipitates inside the grains. TEM study yielded that the material undergoes dynamic recrystallization (DRX) during creep as well as during the FSW process. Various stages of the development of dislocation networks into a cellular dislocation structure and finally into dislocation free recrystallized grains were recorded. The friction stir welded material, which has already recrystallized during welding, undergoes DRX during creep so that ultra-fine grains are being created concurrently. Precipitation processes at the friction

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Rezime

Loša zavarljivost aluminijumske legure AA2024 ograničava njihovu upotrebu u industrijskim primenama. Čini se da je postupak zavarivanja bez topljenja, trenjem sa mešanjem (FSW) obećavajuće rešenje za zavarivanje ove legure. FSW je primenjen u ovoj studiji za zavarivanje ploče od aluminijumske legure AA2024-T3 i kako bi se proučilo puzanje šava. Ispitivanja puzanja su obavljena na 250 °C i 315 °C, na osnovnom materijalu i na epruvetama zavarenim trenjem sa mešanjem. Sveobuhvatna transmisiona elektronska mikroskopija (TEM) zajedno sa skeniranjem elektronskim mikroskopom visoke rezolucije (HRSEM) i analizom rendgenske spektroskopije sa energetsom disperzijom (EDS) sprovedene su u cilju ispitivanja mikrostrukturnih procesa. Čini se da osnovni materijal sadrži dve vrste taloga koji sadrže Cu - grubi talozi veličine nekoliko mikrona i jednoliko dispergovane sitne taloge nanovelicine. Međutim, za ovu mikrostrukturu je utvrđeno da je nestabilna u temperaturnom opsegu od 250 do 315°C, postojali su sekundarni talozi, ovi sekundarni talozi su odgovorni za dekoraciju granica zrna i pojavu sekundarnih taloga u obliku štapa i za određeni stepen ogrubljenja taloga nanovelicine u zrnju. TEM je pokazao da se materijal podvrgava dinamičkoj rekristalizaciji (DRX) tokom puzanja, kao i tokom FSW procesa. Zabeležene su različite faze razvoja dislokacionih mreža u ćelijsku dislokacionu strukturu i konačno u rekristalizovana zrna bez dislokacija. Materijal zavaren trenjem sa mešanjem koji se već rekristalizirao tokom zavarivanja, podvrgnut je DRX-u tokom puzanja,



stir welded material occur as well during creep. The instability of the microstructure during creep and exposure to high temperature plays an important role in the analysis of the creep results. The influence of the above microstructure changes occurring during creep on the creep behavior will be referred and discussed.

1. Introduction

The 2024 (Al-4%Cu-1.5%Mg) aluminum alloy is one of the most widely used materials for airplane structures [1-3], and as such has been investigated in depth to clarify the relationships between its microstructure and its mechanical properties. Although Al-Cu-Mg alloys cannot be considered new materials since their early applications date back to World War I, the 2024 is still the reference material for aerospace and has continuously been improved, with Ag addition to its composition being only one of the most recent developments [4,5]. The creep response of the 2024 alloy has been investigated in detail, with one of the first studies dating back to the late 1950s [6]. More recently, the same alloy was the subject of detailed mechanical and microstructural investigations aimed at correlating the microstructure evolution and the creep response [7-9]. Although at first glance one could legitimately conclude that this material is now fully characterized, many researchers continue to investigate its high temperature response [10-13]. The precipitate study of AA2024 continues to this day. Among other publications dealing with precipitation study, Wang and Starink [14] and Zhang et al. [15] reported new characterized precipitates. Researchers seem to agree that two aging sequences take place. The first is the aging sequence:



while the second is the S aging sequence:



Both sequences begin with a Super Saturated Solid Solution (SSSS) and end with stable precipitates.

tako da se istovremeno stvaraju ultra-fina zrna. Proces taloženja na materijalu koji je zavaren trenjem sa mešanjem, odvija se i tokom puzanja. Nestabilnost mikrostrukture tokom puzanja i izloženost visokoj temperaturi igra važnu ulogu u analizi rezultata puzanja. Uticaj gore navedenih promena mikrostrukture koje nastaju tokom puzanja biće prikazan i razmatran.

1. Uvod

Aluminijska legura 2024 (Al-4% Cu-1,5% Mg) jedan je od najčešće korišćenih materijala za avionske konstrukcije [1-3], i kao takva je detaljno istražena kako bi se razjasnila veza između njene mikrostrukture i mehaničkih svojstava. Iako se legure Al-Cu-Mg ne mogu smatrati novim materijalima, jer njihova rana primena datira još iz Prvog svetskog rata, legura 2024 je još uvek referentni materijal za vazduhoplovstvo i kontinuirano se unapređuje, uz dodatak Ag u njen sastav samo jedan od najvažnijih nedavnih dešavanja [4,5].

Odgovor legura 2024 na puzanje detaljno je istražen, a jedno od prvih istraživanja datira iz kasnih 1950-ih [6]. U novije vreme, ista legura je bila predmet detaljnih mehaničkih i mikrostrukturnih istraživanja usmerenih na povezanost razvoja mikrostrukture i puzanja [7-9]. Iako se na prvi pogled s pravom može zaključiti da je ovaj materijal u potpunosti okarakterisan, mnogi istraživači nastavljaju da istražuju njegovo ponašanje na visokim temperaturama [10-13]. Studija o taloženju AA2024 nastavlja se do danas. Između ostalih publikacija koje se bave proučavanjem taloga, Vang i Starink [14] i Zhang i dr. [15] izvestio je o novim karakterističnim talozima. Čini se da se istraživači slažu sa tim da se odvijaju dve sekvence starenja. Prva sekvenca starenja Θ je:



dok je druga sekvenca starenja S



Obe sekvence počinju super zasićenim čvrstim rastvorom (SSSS) i završavaju se stabilnim talozima.



The poor weldability of the 2024 aluminum alloy using arc welding processes limits the use of the material for industrial applications. Friction Stir Welding (FSW), on the other hand, seems to be a promising solution for welding this alloy. Several studies have used Transmission Electron Microscopy (TEM) to investigate the microstructural changes occurring during FSW of 2024 [16-21]. Leal and Loureiro [16] conducted a dislocation study on friction stir welded 6063-T6 aluminum alloy and reported on the formation of a cellular structure at the nugget zone as opposed to the Thermo Mechanically Affected Zone (TMAZ). While they studied 6063-T6 aluminum alloy, their work is nevertheless mentioned here due to the importance of their TEM study of the Al matrix. Fu et al. [17] investigated the effect of different heat input conditions on grain size during FSW of 2024-T3 aluminum alloy. They reported on dynamic recrystallization (DRX) and subgrain formation at relatively low heat inputs and on extensive grain growth at higher heat inputs. Chen et al. [18] came to the same conclusion regarding grain size. They also pointed to the dissolution of the strengthening precipitates at high welding heat input together with re-precipitation. Dixit et al. [19] referred to precipitation of S (Al₂CuMg) phase precipitates together with the formation of dislocation structures, subgrains and the occurrence of recrystallization processes within the nugget zone. Jones et al. [20] reported on fine equiaxed grains (4-5 μm) at the nugget zone, with some containing high dislocation density while others appearing to have low densities. The grains of the nugget were found to contain two types of precipitates fine scale S precipitates and larger f₂ (Al₂Cu) precipitates. Genevois et al. [21] identified three different dislocation structures: The Heat Affected Zone (HAZ) close to the base metal was characterized by dislocation density similar to that of the base metal, while at the HAZ close to the TMAZ the dislocation density was found to be relatively high, similar to that of the TMAZ in which a network structure was observed. In the nugget, where deformation during welding was maximal, few dislocations remained after welding due to DRX.

In summary, different researchers seem to agree that DRX occurs during friction stir welding of AA2024-T3. The current paper examines the creep of a complex microstructure that underwent extensive plastic deformation, in turn yielding recrystallization prior to creep (during welding). In addition, the AA2024-T3 is known to undergo different precipitation and aging stages. To the best knowledge of the authors, no model for creep of

Loša zavarljivost aluminijumske legure 2024 elektrolučnim postupcima zavarivanja ograničava upotrebu materijala za industrijske namene. Čini se da je zavarivanje trenjem sa mešanjem (FSW) obećavajuće rešenje za zavarivanje ove legure. Nekoliko studija koristi transmisionu elektronsku mikroskopiju (TEM) za istraživanje mikrostrukturnih promena koje su se dogodile tokom FSW legure 2024 [16-21]. Leal i Loureiro [16] sproveli su dislokacijsku studiju na aluminijskoj leguri 6063-T6 zavarenoj trenjem sa mešanjem i izvestili o formiranju ćelijske strukture u zoni gromuljice, za razliku od termo mehanički zahvaćene zone (TMAZ). Dok su proučavali leguru aluminijuma 6063-T6, njihov rad se ovde ipak spominje zbog važnosti njihove TEM studije Al matrice. Fu i dr. [17] istražio je uticaj različitih uslova toplote na veličinu zrna tokom FSW aluminijumske legure 2024-T3. Izveštavali su o dinamičkoj rekristalizaciji (DRX) i stvaranju subzrna pri relativno malim unosima toplote i o ekstenzivnom rastu zrna pri većim unosima toplote. Chen i dr. [18] došli su do istog zaključka u pogledu veličine zrna. Takođe su ukazali na rastvaranje taloga za ojačanje pri visokom unosu toplote pri zavarivanju, zajedno sa ponovnim taloženjem. Dikit i dr. [19] saopštavaju o taloženju taloga S (Al₂CuMg) faze zajedno sa formiranjem dislokacionih struktura, subzrna i pojavom procesa rekristalizacije u zoni izrasline. Jones i dr. [20] izveštavali su o finim istoosnim zrcima (4-5 μm) u zoni izrasline, pri čemu neki imaju veliku gustinu dislokacija, dok drugi izgleda da imaju malu gustinu. Otkriveno je da zrno u izbočini sadrži dve vrste taloga; sitnih taloga S i više f₂ (Al₂Cu) taloga. Genevois i dr. [21] identifikovali su tri različite dislokacione strukture: Zona uticaja toplotne energije (HAZ) blizu osnovnog metala bila je okarakterisana gustinom dislokacije sličnom kao kod osnovnog metala, dok je kod HAZ-a blizu TMAZ-a gustina dislokacija relativno visoka, slično kao kod TMAZ-a u kome je primećena mrežna struktura. U izbočini, gde je deformacija tokom zavarivanja bila maksimalna, nakon zavarivanja zbog DRX ostalo je nekoliko dislokacija.

Ukratko, čini se da se različiti istraživači slažu da se DRX javlja tokom zavarivanja trenjem sa mešanjem legure AA2024-T3. Sadašnji rad ispituje puzanje složene mikrostrukture koja je pretrpela veliku plastičnu deformaciju, zauzvrat dajući rekristalizaciju pre puzanja (tokom zavarivanja). Pored toga, poznato je da AA2024-T3 prolazi kroz različite faze taloženja i starenja. Koliko je poznato



such a complex microstructure has ever been proposed.

2. Experimental procedure

The material used for this study was commercial AA2024- T3 aluminum alloy in the form of 200 mm x100 mm plates, 3.175 mm thick. The above plates were butt welded to each other using a SHARNOA CNC milling machine. The simple H-13 steel welding tool used consisted of a pin of 4.5 mm diameter, 3 mm height and a 20 mm diameter shoulder.

All the welded specimens were visually examined. Metallographic specimens were extracted from the welds characterized by proper morphology. Four metallographic specimens were prepared from each of them. The first metallographic specimen was taken from the first quarter of the seam, the second from the second quarter and so on, in order to detect inner porosity or cracking. The welds found to be of the highest quality based on metallographic cross- sections were then radiographically checked. The optimal welding parameters were found to be a rotational speed of 800 rpm and a transverse speed of 80 mm/min. The metallographic study was conducted using a Zeiss AX10 optical microscope and a Zeiss Ultra Plus High Resolution Scanning Electron Microscope (HRSEM).

The configuration of the creep specimens with respect to the welded specimen is shown in figure 1a, while its dimensions are given in Fig. 1b. The gage length was 25 mm, with the center of the weld seam corresponding to the center of the creep specimen. Note that the gage length included the weld nugget, the TMAZs, the HAZs and the parent materials on both sides of the weld.

autorima, nikada nije predložen model za puzanje tako složene mikrostrukture.

2. Eksperimentalni postupak

Materijal korišćen za ovu studiju je komercijalna legura aluminija AA2024-T3 u obliku ploča od 200 x 100 mm, debljine 3.175 mm. Gore navedene ploče su međusobno zavarene pomoću glodalice SHARNOA CNC. Jednostavan alat za zavarivanje od čelika H-13 sastojao se od igle prečnika 4,5 mm, visine 3 mm i prečnika držača od 20 mm.

Svi zavareni uzorci su vizuelno pregledani. Metalografski uzorci su uzeti iz šavova koje karakteriše odgovarajuća morfologija. Iz svakog od njih pripremljena su četiri metalografska uzorka. Prvi metalografski uzorak uzet je iz prve četvrtine šava, drugi iz druge četvrtine i tako dalje, da bi se otkrila unutrašnja poroznost ili prslina. Pronađeni su najkvalitetniji zavari na osnovu metalografskih preseka, a zatim su radiografski pregledani. Otkriveni su optimalni parametri zavarivanja, brzina rotacije od 800 o/min i poprečna brzina od 80 mm/min. Metalografska studija izvedena je uz pomoć optičkog mikroskopa Zeiss AKS10 i Zeiss Ultra Plus skenirajućeg elektronskog mikroskopa (HRSEM).

Konfiguracija uzoraka za puzanje u odnosu na zavarene epruvete prikazana je na slici 1a, dok su njene dimenzije date na slici 1b. Dužina merenja je 25 mm, a sredina zavarenog šava odgovara sredini uzorka puzanja. Imajte na umu da je dužina merača obuhvatala izbočinu, TMAZ-ove, HAZ-ove i osnovni materijal sa obe strane šava.

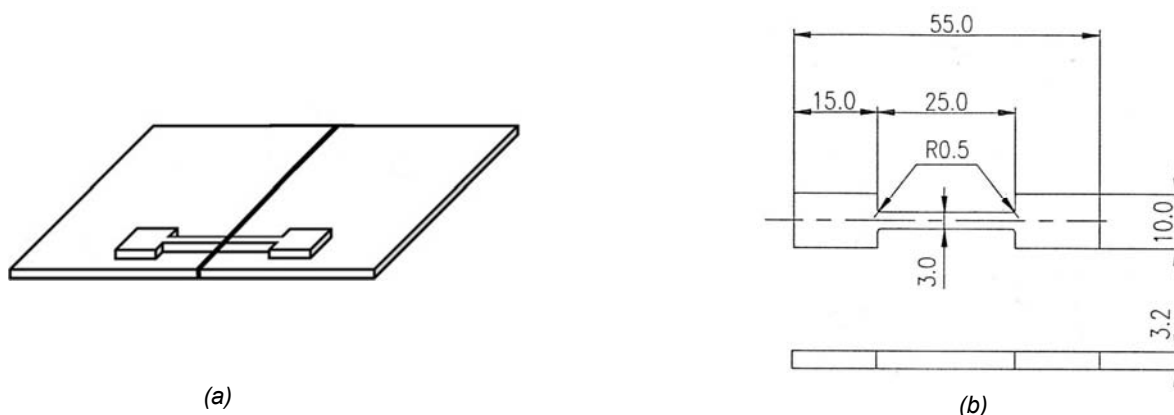


Fig. 1 Creep specimen (a) Configuration; (b) A drawing
Sl.1. Epruveta za puzanje (a) konfiguracija (b) crtež



Constant load creep tests were conducted on the base alloy (CLb) and on the cross-welds (CLcw) at 250°C and at 315°C, in most cases up to fracture. The experimental temperature, which was well above the maximum allowable temperature usually prescribed for the 2024 alloy, was specifically chosen to facilitate studying the effects of microstructural instability on the creep response. To achieve the same goal, additional variable load experiments were carried out on the base alloy (CLcw). In these experiments, the initial stress (25 MPa, except in one experiment in which the initial stress was 15 MPa) was maintained until the minimum creep rate range was attained. The test duration required to reach the minimum creep rate range was estimated based on the results of CLb, which had previously been carried out under the corresponding stress. The applied stress was then abruptly increased and maintained up to specimen rupture. The gage-length of the cross-weld specimens was marked by micro-hardness indentations distanced 1mm from one another. After creep, the distance between the indentations was measured to evaluate the strain distribution along the specimen.

TEM investigation was conducted by using an FEI Tecnai G° T20 TEM. The specimens for the TEM study were taken from the neck of the broken creep specimens, as close as possible to the fracture surface.

The thermal stability of the microstructure was studied by means of aging experiments conducted at 300°C for up to 280 hours.

3. Results

Fig. 2 shows an optical micrograph of the parent AA2024-T3 taken parallel to the rolling direction. In the figure, the grains are elongated due to the rolling process.

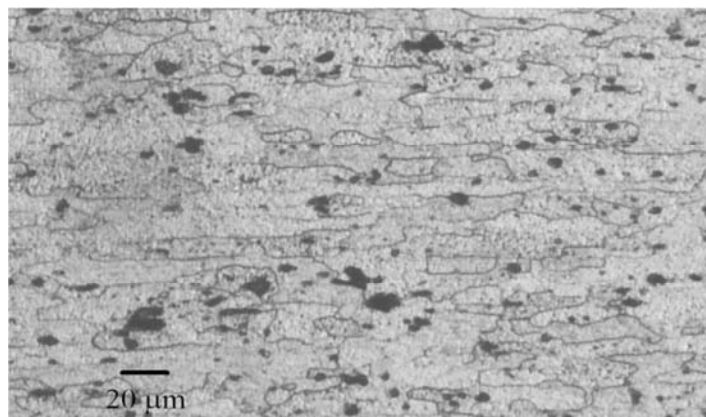


Fig. 2 An optical micrograph of the parent AA2024-T3
Sl.2. Optička mikrofografija osnovnog materijala A2024-T3

Ispitivanja puzanja konstantnim opterećenjem izvedena su na osnovnoj leguri (CLb) i na poprečnim zavarima (CLcv) na 250°C i na 315 ° C, u većini slučajeva do loma. Eksperimentalna temperatura, koja je bila znatno iznad maksimalne dozvoljene temperature, obično propisane za leguru 2024, posebno je odabrana da olakša proučavanje efekata mikrostrukturne nestabilnosti na reakciju na puzanje. Da bi se postigao isti cilj, obavljani su dodatni eksperimenti sa promenljivim opterećenjem na osnovnoj leguri (CLcv). U tim eksperimentima je inicijalni napon (25 MPa, osim u jednom eksperimentu u kome je početni napon bio 15 MPa) održavan sve dok se nije dostigao raspon minimalne brzine puzanja. Trajanje testa potrebno da se dostigne raspon minimalne brzine puzanja procenjeno je na osnovu rezultata CLb, koji su prethodno izvedeni pod odgovarajućim naponom. Primenjeni napon je naglo porastao i održavao se sve do pucanja uzorka. Debljina dužine ukrštenih uzoraka bila je obeležena udubljenjima mikrotvrdoće udaljenim mm od jednog do drugog. Nakon puzanja, izmerena je udaljenost između udubljenja kako bi se procenila raspodela naprezanja po uzorku.

TEM istraživanje je sprovedeno korišćenjem FEI Tecnai G ° T20 TEM. Uzorci za TEM ispitivanje uzeti su sa vrata slomljenih uzoraka, što bliže površini preloma.

Termička stabilnost mikrostrukture proučavana je eksperimentima starenja vođenim na 300 ° C tokom 280 sati.

3. Rezultati

Sl. 2 prikazuje optičku mikrofografiju osnovne legure AA2024-T3 uzete paralelno sa pravcem valjanja. Na slici su zrna izdužena zbog procesa valjanja.



Fig. 3 shows an optical micrograph taken from the nugget zone. In contrast to the elongated grains in figure 2, the grains seen in figure 3 are equi-axed and their average size is a few microns each.

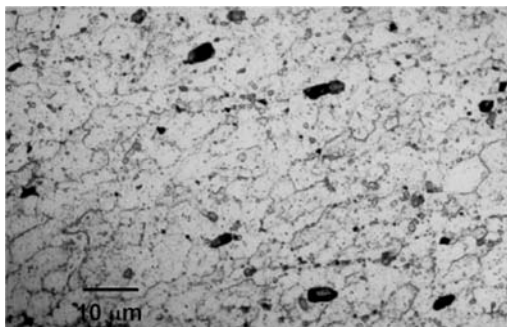


Fig. 3 An optical micrograph of the nugget zone of friction stir welded AA2024-T3
Sl. 3. Optička mikrografija izbočene zone FSW AA2024-T3

Micro hardness tests showed that the hardness of the material as received was 100-110 HV. Despite the grain refinement, the FSW process caused only a moderate increase in hardness, which reached its maximum values (130 HV) at the center of the nugget zone and dropped down to 95-100 HV, i.e., to values slightly lower than the base metal, in the heat affected zones.

Figure 4 shows the dependence of the minimum creep rate on the applied stress for the CLb, VLb and CLcw experiments. The CLb results lie along a straight line with a slope of 4.4. In contrast, the slope of the curve of the VLb experiments is substantially higher than 4.4. Fig. 4 also plots the minimum creep rate of the cross weld samples after FSW. The figure clearly shows that the FSW samples exhibited a minimum creep rate that is higher by orders of magnitude than that of the base alloy, tested either under constant or variable load.

Testovi mikro tvrdoće pokazali su da je tvrdoća materijala koji je primljene 100-110 HV. I pored rafinacije zrna, FS>W proces je izazvao samo umereno povećanje tvrdoće koja je dostigla svoje maksimalne vrednosti (130 HV) u središtu izbočene zone i spustila se do 95-100 HV, tj. vrednosti nešto niže od osnovnog materijala, u zonama pod uticajem toplote.

Slika 4 prikazuje zavisnost minimalne brzine puzanja o primenjenom stresu za CLb, VLb i CLcw eksperimente. Rezultati CLb leže duž ravne linije sa nagibom od 4,4. Suprotno tome, nagib krivulje VLb eksperimenata znatno je veći od 4,4. Sl. 4 takođe prikazuje minimalnu brzinu puzanja uzoraka unakrsnog zavarivanja nakon FSV. Na slici je jasno vidljivo da su uzorci FSV pokazali najmanju brzinu puzanja koja je veća za redosled veličine od stope osnovne legure, testirane ili pod konstantnim ili promenljivim opterećenjem.

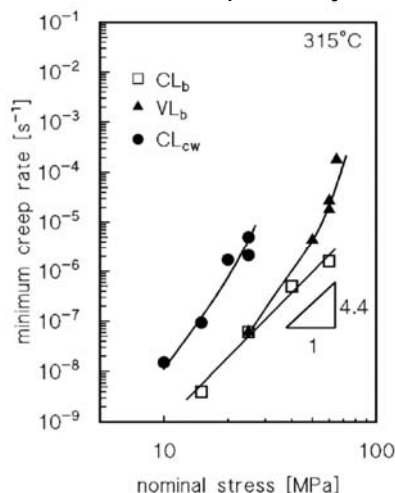


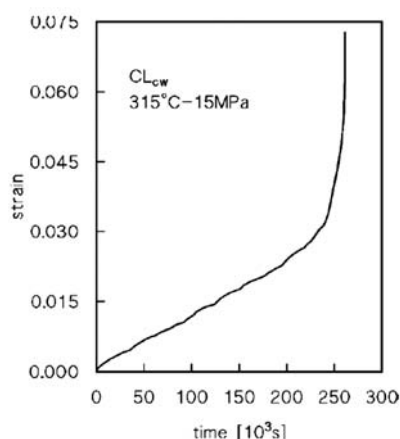
Fig. 4 The minimum creep rate as a function of the nominal testing stress for the base alloy and for the cross-welded FSW samples

Sl. 4 Minimalna brzina puzanja kao funkcija nominalnog ispitnog napreznja za baznu leguru i za poprečno zavarene FSW uzorke

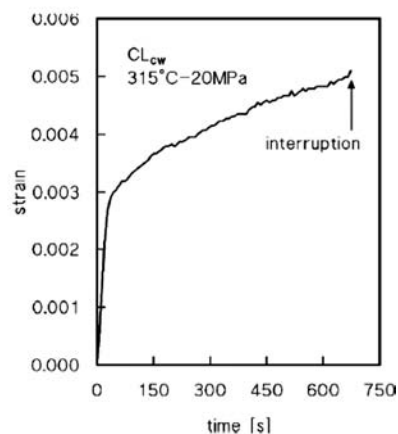


Fig. 5 shows two creep curves of friction stir welded specimens. Curve (a) refers to a test carried out at 3150C under 15MPa. This test was continued until rupture. Curve (b) refers to a test carried out at 315 °C under 20 MPa, which was interrupted when the minimum creep rate was reached, prior to the tertiary stage.

Sl. 5 prikazuje dve krivine puzanja uzoraka zavarivanih trenjem pomoću mešavina trenja. Krivulja (a) se odnosi na test sproveden na 3150C ispod 15MPa. Ovaj test je nastavljen do puknuća. Kriva (b) odnosi se na test sproveden na 315 °C ispod 20 MPa, koji je prekinut kada je dostignuta minimalna brzina puzanja, pre tercijarne faze.



a)



b)

Fig. 5 Creep curves for tests carried out on cross-weld FSW samples (a) Until rupture; (b) Interrupted
Sl. 5 Krive puzanja za ispitivanja izvedena na uzorcima unakrsnog zavarivanja FSW (a) Do prekida; (b) Prekinuto

Fig. 6 shows an optical micrograph of the creep specimen after the test was interrupted. An extended 1.4 mm long crack clearly appears at the weld root. Similar cracks were found in all the FSW crept samples. Micro-hardness indentations created on the creep specimen prior to the test at a constant spacing of 1 mm showed that most of the gage length deformation occurred at a 2 mm long interval in the vicinity of the crack. This led to the conclusion that rupture was accompanied by extensive localized deformation.

Sl. 6 prikazuje optičku mikrografiju uzorka za puzanje nakon što se test pokrenuo. Proširena pukotina dužine 1,4 mm jasno se pojavljuje na korenu zavora. Slične prsline pronađene su u svim uzorcima FSW, podvrgnutim starenju. Udubljena mikro tvrdoće stvorena na uzorku za puzanje, pre ispitivanja, pri konstantnom razmaku od 1 mm pokazala su da se najveći deo deformacije merača dogodio u razmaku od 2 mm u blizini prsline. To je dovelo do zaključka da je lom praćen obimnom lokalizovanom deformacijom.

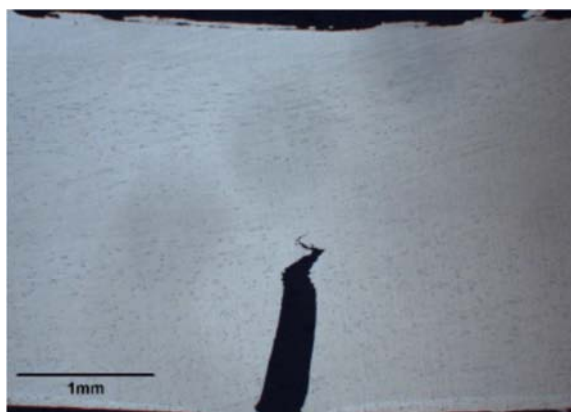
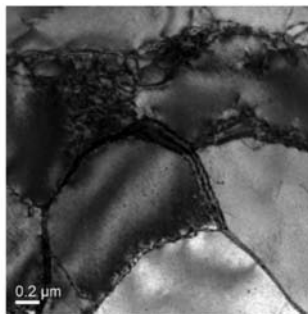


Fig. 6 An optical micrograph of the creep specimen after interruption (25 MPa, 315 C)
Fig. 6 An optical micrograph of the creep specimen after interruption (25 MPa, 315 C)

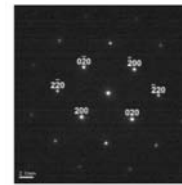


A Bright Field (BF) TEM micrograph of AA2024-T3 parent material taken near $\langle 001 \rangle$ Z.A is shown in figure 7, while a selected area electron diffraction pattern of $\langle 001 \rangle$ Z.A is given in Fig. 7b. A sub-grain structure whose boundaries consist of dislocation networks can be clearly identified in this micrograph. Moreover, as known, when a crystal is tilted into a certain zone axis, its bright field becomes darker because more energy goes to the diffracted beams instead of to the incident beam. Here the dark contrast proves that all three are near $\langle 001 \rangle$ Z.A., as can be expected in the case of sub-grains as opposed to grains. The size of these sub-grains is a few microns each.

Na slici 7 prikazana je TEM mikrografija osnovnog materijala AA2024-T3 sa svetlim poljem (BF), dok je odabrani dijagram difrakcije elektrona od $\langle 001 \rangle$ Z.A dat na slici 7b. Sstruktura subzrna čije se granice sastoje od dislokacijskih mreža može se jasno identifikovati u ovoj mikrografiji. Štaviše, kao što je poznato, kad se kristal nagne u određenu zonu ose, njegovo svetlo polje postaje tamnije jer više energije odlazi na difraktovane zrake, umesto na padajući snop. Ovde tamni kontrast dokazuje da su sva tri blizu $\langle 001 \rangle$ Z.A., kao što se može očekivati u slučaju subzrna, za razliku od zrna. Veličina ovih subzrna je nekoliko mikrona.



a)



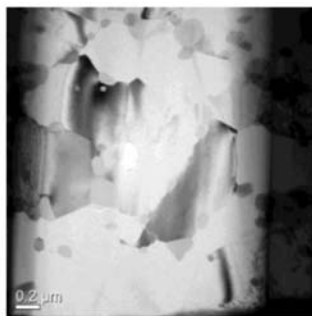
b)

Fig.7 (a) BF TEM micrograph of AA2024-T3 parent material taken near $\langle 001 \rangle$ Z.A.; (b) Selected area electron diffraction pattern of $\langle 001 \rangle$ Z.A

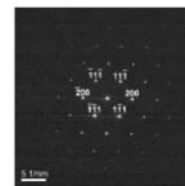
Sl. 7 (a) BF TEM mikrografija osnovnog materijala AA2024-T3 snimljena blizu $\langle 001 \rangle$ Z.A. ; (b) Odabrani elektronski difrakcijski oblik $\langle 001 \rangle$ Z.A

Fig. 8a shows a BF TEM micrograph of AA2024-T3 after creeping 17 hrs at 250 °C under 120MPa taken near $\langle 011 \rangle$ Z.A., while a selected area electron diffraction pattern of $\langle 011 \rangle$ Z.A is shown in Fig. 8b. The dislocation networks seen in figure 7 are not discernible in figure 8. The dark contrast of the grains tilted to $\langle 011 \rangle$ Z.A makes it possible to identify them and to estimate their size. It seems that there are many submicron grains, and the appearance of such ultrafine single grains can be related to DRX. Note that the sub-grain structure almost disappeared.

Sl. 8a prikazuje BF TEM mikrografiju AA2024-T3 nakon puzanja 17 sati na 250 ° C pod 120MPa snimljenom blizu $\langle 011 \rangle$ Z.A., dok je odabrani difrakcijski elektronski obrazac $\langle 011 \rangle$ Z.A prikazan na slici 8b. Mreže dislokacije prikazane na slici 7 nisu prepoznatljive na slici 8. Tamni kontrast zrna nagnutog ka $\langle 011 \rangle$ Z A omogućava da se identifikuje i proceni njihova veličina. Čini se da postoji mnogo subzrna , a pojava takvih ultrafinih pojedinačnih zrna može biti povezana sa DRX-om. Treba zapaziti da da je struktura subzrna gotovo nestala.



a)



b)

Fig. 8 (a) BF TEM micrograph of AA2024-T3 after creeping 17 hrs at 250°C under 120MPa taken near $\langle 011 \rangle$ Z.A.; (b) Selected area electron diffraction pattern of $\langle 011 \rangle$ Z.A

Sl. 8 (a) BF TEM mikrografija AA2024-T3 posle puzanja 17 sati na 250 ° C pod 120MPa, snimljena blizu $\langle 011 \rangle$ Z.A. ; (b) Izabrani elektronski difrakcijski oblik $\langle 011 \rangle$ Z.A



Fig. 9 depict BF TEM micrographs of the friction stir welded AA2024-T3 in its as-weld condition. No evidence of sub-grain structure was detected. For example, Fig. 9a was taken after tilting the right grain to $\langle 001 \rangle$ Z.A. Its selected area electron diffraction pattern is shown in Fig. 9b. Tilting the left grain to $\langle 001 \rangle$ Z.A. (Fig. 9d) required a large tilt angle. This shows, in turn, that these are two different grains, each a few microns in size.

Sl. 9 prikazuju BF TEM mikrografije AA2024-T3 zavarenog trenjem sa mešanjem, u stanju zavarivanja. Nisu otkriveni dokazi o strukturi subzrna. Na primer, slika 9a je uzeta nakon nagiba desnog zrna do $\langle 001 \rangle$ Z.A. Njegov odabrani oblik difrakcije elektrona za to područje prikazan je na slici 9b. Naginjanje levog zrna do $\langle 001 \rangle$ Z.A. (Sl. 9d) zahteva veliki ugao nagiba. To pokazuje, sa svoje strane, da su to dva različita zrna, svaka veličine nekoliko mikrona.

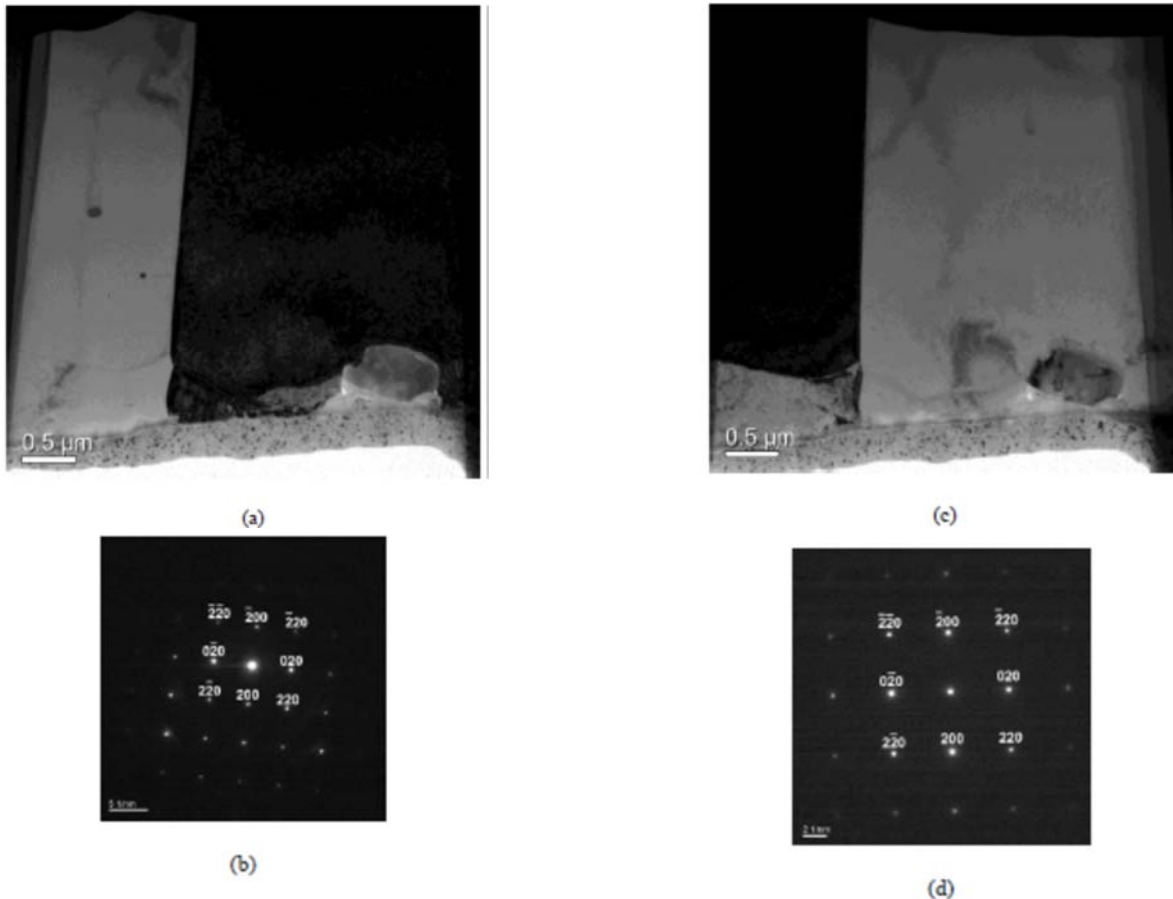


Fig. 9 (a, c) BF TEM micrograph of friction stir welded AA2024-T3 taken while the dark grains were tilted to $\langle 001 \rangle$ Z.A.; (b, d) Selected area electron diffraction patterns of $\langle 001 \rangle$ Z.A taken from each grain

Sl. 9 (a, c) BF TEM mikrografija AA2024-T3 zavarenog trenjem sa mešanjem, snimljena dok su tamna zrna nagnuta ka $\langle 001 \rangle$ Z.A. ; (b, d) Izabrani uzorci elektronske difrakcije elektrona $\langle 001 \rangle$ Z.A uzeti iz svakog zrna

Figure 10a shows a BF TEM micrograph of friction stir welded AA2024-T3 after creeping 139 hrs at 3150C under 120MPa. The dark grain was tilted to $\langle 013 \rangle$ Z.A. Its selected area electron diffraction pattern is shown in Fig. 10b, while the selected area electron diffraction pattern of the neighboring grain on its right is depicted in Fig.10c.

It can be concluded that these are two separate grains, each a few microns in size. Finer grains can be seen in Fig. 10a as well.

Na slici 10a prikazana je BF TEM mikrografija AA2024-T3 zavarenog trenjem sa mešanjem, nakon puzanja 139 sati na 315 ° C pod 120MPa. Tamno zrno je nagnuto ka $\langle 013 \rangle$ Z.A. Njegov odabrani oblik područja difrakcije elektrona prikazan je na slici 10b, dok je odabrani dijagram difrakcije elektrona susednog zrna sa desne strane prikazan na slici 10c.

Može se zaključiti da su to dva odvojena zrna, svaka veličine nekoliko mikrona. Finija zrna mogu se videti i na slici 10a.

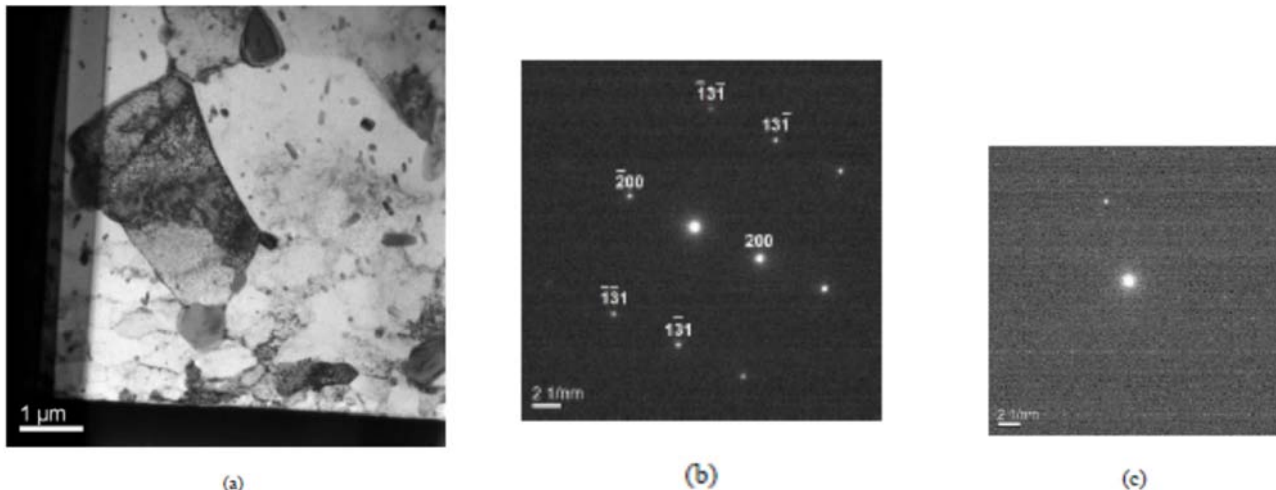


Fig. 10 (a) BF TEM micrograph of friction stir welded AA2024-T3 after creeping 139 hrs at 315°C; (b) Selected area electron diffraction pattern of $\langle 013 \rangle$ Z.A — dark grain; (c) Selected area electron diffraction pattern taken from the neighboring grain

Sl. 10 (a) BF TEM mikrografija AA2024-T3 zavarenog trenjem sa mešanjem, nakon puzanja 139 sati na 315 °C; (b) Odabrani oblik difrakcije elektrona $\langle 013 \rangle$ Z.A - tamno zrno; (c) Odabrani dijagram difrakcije elektrona odabranog područja iz susjednog zrna

Fig. 11 shows two HRSEM images. Fig. 11a refers to the as-received parent metal, while figure 11 b was taken after 170 hours of exposure to 315 °C. Coarse precipitates a few microns in size are discernible in the as-received material, together with evenly dispersed nano-sized precipitates. The aged material (Fig. 11b) reveals two other types of precipitates. The first type of precipitates decorates the grain boundaries, while the second type contains platelet-like or rod-shaped precipitates. Coarsening to some degree of the nano-sized precipitates inside the grains can be seen as well.

Sl. 11 prikazuje dve HRSEM slike. Sl. 11a odnosi se na osnovni metal u isporučenom stanju, dok je slika 11b uzeta nakon 170 sati izloženosti na 315 °C. Grubi talozi veličine nekoliko mikrona mogu se primetiti po primljenom materijalu, zajedno sa ravnomerno dispergovanim talogom nano veličine. Stareni materijal (Sl. 11b) otkriva dve druge vrste taloga. Prva vrsta taloga dekoriše granice zrna, dok druga vrsta sadrži u obliku trombocita ili šipke. Takođe se može videti grubo izlučeni talog nano veličine u zrnu.

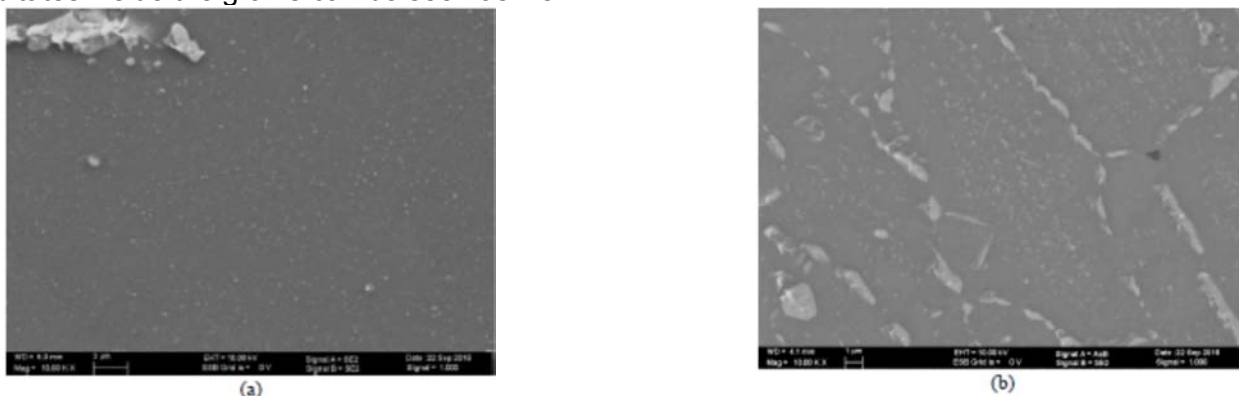


Fig. 11 HRSEM images of (a) As-received material (b) After 170 hour long exposure to 315 °C
Sl. 11 HRTEM slike (a) Primljeni materijal (b) Posle 170 sati dugog izlaganja na 315 °C

Systematic precipitation analysis conducted using both TEM and HRSEM indicated that these precipitates can either contain Al, Cu and Mg or can contain just Al and Cu. Examples of such precipitates are given in Fig. 12.

Sistematska analiza taloga sprovedena korišćenjem i TEM i HRSEM pokazala je da ovi talozi mogu da sadrže Al, Cu i Mg ili mogu da sadrže samo Al i Cu. Primeri takvih taloga su dati na slici 12.

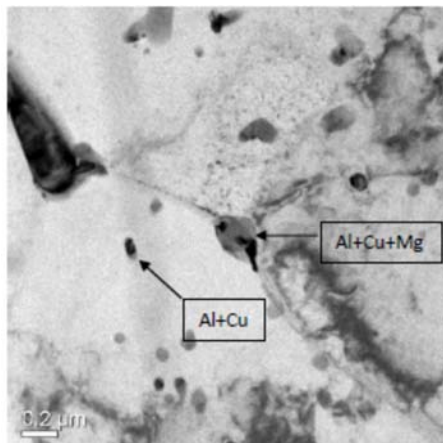


Fig. 12 BF TEM image of a specimen exposed to 315 °C for 139 hrs
Sl. 12 BF TEM slika uzorka izložena 315 °C tokom 139 sati

4. Discussion

As stated earlier clearly shows that the FSW samples exhibited a minimum creep rate that is higher by orders of magnitude than that of the base alloy, tested either under constant or variable load. The stress exponent calculated, namely 4.4, points at dislocation creep, however, the current study brings clear evidence of the microstructure instability during creep. TEM study yielded that high dislocation density and networks were observed at the parent metal (see Fig. 8) while dislocation free grains at the welded nugget (see Fig. 10) point at DRX occurring due to heavy plastic deformation during FSW as stated by Genevois et al. [2] and other researchers [17,19]. This conclusion is in line with optical microscopy results. The appearance of submicron dislocation free grains during creep (see Fig. 9) may point also at DRX processes occurring during creep. The material contains in its as received condition coarse precipitates together with evenly dispersed nano-sized ones as can be seen in Fig. 12a, keeping in mind that the thermal condition of the alloy is T3, namely, solution heat treated, cold worked, and naturally aged, the primary coarse precipitates can be due to insufficient solution treatment. Secondary precipitation processes occur then during exposure to creep temperature, this secondary precipitation results in grain boundary decoration, appearance of secondary rod-shaped precipitates and some degree of coarsening of the nano sized precipitates inside the grains. Aging experiments and chemical analysis of the precipitates showed that the secondary precipitates are of both types Al-Cu-Mg and Al-Cu, it can be concluded, therefore, that both S and aging sequences took place. As for the friction stir welded specimens, the situation is even more complicated in that case because the material which has already recrystallized during welding

4. Diskusija

Kao što je ranije navedeno, jasno je vidljivo da su uzorci FSW pokazali minimalnu brzinu puzanja koja je veća za red veličine od one u osnovnom materijalu, testirano ili pod konstantnim ili promenljivim opterećenjem. Izračunati eksponent napona, tačke.4,4 pri puzanju dislokacije, međutim, trenutna studija donosi jasne dokaze o nestabilnosti mikrostrukture tokom puzanja. TEM studija je pokazala da su na osnovnom materijalu primećene visoke gustine dislokacije i mreže (vidi Sl. 8), dok se zrna bez dislokacije u izbočini zavarenoj spoja (vidi Sl. 10) pri DRX-u nastaju usled teške plastične deformacije tokom FSW-a kako su naveli Genevois I dr. [2] i drugi istraživači [17,19]. Ovaj zaključak je u skladu s rezultatima optičke mikroskopije. Pojava zrna bez submikronskih dislokacije tokom puzanja (vidi Sliku 9) može ukazivati i pri DRX na procese koji se javljaju tokom puzanja. Materijal sadrži u svom primljenom stanju grube taloge zajedno sa ravnomerno raspodeljenim nano veličine kao što se vidi na slici 12a, imajući u vidu da je termičko stanje legure T3, naime, obrađen termičkim rastvaranjem,, hladno obrađen, i prirodno staren, primarni grubi talozi mogu biti posledica nedovoljnog tretmana rastvora. Postupci sekundarnog taloženja se javljaju tokom izlaganja temperaturi puzanja, ti sekundarni talozi rezultiraju dekorom na granici zrna, pojavom sekundarnih taloga u obliku štapa i određenim stepenom ogrubljenja taloga nano veličine unutar zrna. Eksperimenti starenja i hemijska analiza taloga, pokazali su da su sekundarni talozi , obe vrste Al-Cu-Mg i Al-Cu, pa se može zaključiti da i S i starenje zauzimaju svoje mesto. Što se tiče zavarenih uzoraka trenjem sa mešanjem, situacija je u tom slučaju još složenija jer se materijal koji je već rekristalizovan tokom zavarivanja podvrgava undergoes DRX



during creep so that ultra- fine grains are being created concurrently, in addition, the secondary precipitation processes mentioned above occur during creep of the welded material as well as in the case of the parent material.

5. Conclusions

- The microstructure of AA2024-T3 was found to be unstable at the temperature range of 250-315°C, secondary precipitation was found to take place, this secondary precipitation is responsible for grain boundary decoration and the appearance
- TEM study yielded that the material undergoes DRX during creep
- The material undergoes DRX during the FSW process
- The friction stir welded material, which has already recrystallized during welding, undergoes DRX during creep so that ultra-fine grains are being created concurrently. Precipitation processes at the friction stir welded material occur as well during the process
- The instability of the micro-structure during creep and exposure to high temperature plays an important role in the analysis of the creep results
- Cracking was found to limit the creep resistance of the friction stir welded AA2024
- Further research work is required in order to eliminate the cracking and improve the creep resistance of the friction stir welded AA2024-T3

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DRX tokom puzanja, tako da se istovremeno stvaraju ultrafina zrna, pored toga, gore navedeni sekundarni procesi taloženja nastaju tokom puzanja zavarenog materijala, kao i kod osnovnog materijala.

5. Zaključci

- Otkriveno je da je mikrostruktura AA2024-T3 nestabilna u temperaturnom opsegu 250-315°C, da su se pojavili sekundarni talozi , a ovi sekundarni talozi su odgovorni za dekor granica zrna i izgled
- TEM proučavanje je pokazalo da se tokom puzanja, materijal podvrgava DRX
- Materijal je podvrgnut DRX tokom FSW procesa
- Materijal zavaren trenjem sa mešanjem koji je već rekristalizovao tokom zavarivanja, podvrgava se DRX tokom puzanja tako da se istovremeno stvaraju i ultrafina zrna. Proces taloženja na materijalu za zavarivanje trenjem dešavaju se tokom procesa
- Nestabilnost mikro strukture tokom puzanja i izloženost visokoj temperaturi igraju važnu ulogu u analizi rezultata puzanja.
- Nađene su prsline koje ograničavaju otpornost na puzanje AA2024 zavarenog trenjem
- Potrebni su dodatni istraživački radovi kako bi se uklonile prsline i poboljšala otpornost na puzanje AA2024 zavarenog trenjem

Zahvalnost

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