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(54) **FOUR-STEP THERMAL AGING METHOD FOR IMPROVING ENVIRONMENTALLY ASSISTED CRACKING RESISTANCE OF 7XXX SERIES ALUMINIUM ALLOYS**

VIERSTUFIGES THERMISCHES ALTERUNGSVERFAHREN ZUR VERBESSERUNG DER UMWELTUNTERSTÜTZTEN RISSBESTÄNDIGKEIT VON ALUMINIUMLEGIERUNGEN DER 7XXX-SERIE

PROCÉDÉ DE VIEILLISSEMENT THERMIQUE À QUATRE ÉTAPES POUR L'AMÉLIORATION DE LA RÉSISTANCE À LA FISSURATION INFLUENCÉE PAR L'ENVIRONNEMENT D'ALLIAGES D'ALUMINIUM DE SÉRIE 7XXX

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- **KRISHNAN MAJAY ET AL: "Development of high strength AA 7010 aluminum alloy resistant to environmentally assisted cracking", CORROSION SCIENCE, OXFORD, GB, vol. 109, 25 March 2016 (2016-03-25), pages 94-100, XP029533044, ISSN: 0010-938X, DOI: 10.1016/J.CORSCI.2016.03.025**

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Description

FIELD OF THE INVENTION

[0001] This invention relates to a four-step thermal aging method for improving environmentally assisted cracking resistance of 7xxx series aluminium alloys.

BACKGROUND OF THE INVENTION

[0002] Aluminium alloys find extensive use across a wide range of applications from structures to household appliances. Low cost, light weight, versatility and recyclability are some of the main advantages of aluminium alloys. Various grades of aluminium alloys are available depending mainly on their end use and requirement. AA 7xxx or AA 7000 series aluminium alloys such as AA 7010, AA 7050, and AA 7085 are characterized by their high strength and high environmentally assisted cracking resistance and are extensively used for structural applications in aerospace and armor industries.

[0003] Stress corrosion cracking (SCC) and hydrogen embrittlement (HE), together termed as environmentally assisted cracking (EAC), are the main causes of various catastrophic failures of structures comprising 7xxx series aluminium alloys [M. Bobby Kannan, R Raman, A Mukhopadhyay, V S Raja, Influence of multistep aging on the stress corrosion cracking behavior of aluminum alloy 7010, Corrosion 59 (2003) 881-889; M Bobby Kannan, V S Raja, Role of coarse intermetallic particles on the environmentally assisted cracking behavior of peak aged and over aged Al-Zn-Mg-Cu-Zr alloy during slow strain rate testing, J Mater Sci 42 (2007) 5458-5464; M Bobby Kannan, V S Raja, Enhancing stress corrosion cracking resistance in Al-Zn-Mg-Cu-Zr alloy through inhibiting recrystallization, Eng Fract Mech 77 (2010) 249-256; M Bobby Kannan, V S Raja, Hydrogen embrittlement susceptibility of over aged 7010 Al-alloy, J Mater Sci 41 (2006) 5495-5499; M B Kannan, V S Raja, Enhancing the Localized Corrosion Resistance of High Strength 7010 Al-Alloy, Adv Mater Res 138 (2010) 1-6].

[0004] Chemical composition and quench sensitivity of some of the 7xxx series aluminium alloys are given in the following Table 1:

Table 1

Alloy	Chemical Composition, (wt.%)						Quench Sensitivity	
	Zn	Mg	Cu	Fe	Si	Zr	Al	
AA7010	5.7-6.7	2.1-2.6	1.5-2.0	<0.15	<0.12	0.1-0.16	Bal	High
AA7050	5.7-6.7	1.9-2.6	2-2.6	<0.15	<0.12	0.08-0.15	Bal	Medium
AA7085	7.0-8.0	1.2-1.8	1.3-2.0	<0.08	<0.06	0.08-0.15	Bal	Low

[0005] The 7xxx series alloys are subjected to various thermal aging treatments in order to enhance their strength and EAC resistance. (US4477292, US4431467, US2003/0041934A1, US4477292, US8333853, US9249487 and US2015/0259774A1; M Bobby Kannan, R Raman, A Mukhopadhyay, V S Raja, Influence of multistep aging on the stress corrosion cracking behavior of aluminum alloy 7010, Corrosion 59 (2003) 881-889; T C Tsai, T H Chuang, Atmospheric stress corrosion cracking of a superplastic 7475 aluminum alloy, Metall Mater Trans A 27 (1996) 2617-2627; M F Ibrahim, A M Samuel, F H Samuel, A preliminary study on optimizing the heat treatment of high strength Al-Cu-Mg-Zn alloys, Mater Des. 57 (2014) 342-350; J C Lin, H L Liao, W D Jehng, C H Chang, S L Lee, Effect of heat treatments on the tensile strength and SCC-resistance of AA7050 in an alkaline saline solution, Corros. Sci. 48 (2006) 3139-3156; A K Mukhopadhyay, K S Prasad, V Kumar, G M Reddy, S V Kamat, V K Varma, Key Microstructural Features Responsible for Improved Stress Corrosion Cracking Resistance and Weldability in 7xxx Series Al Alloys Containing Micro / Trace Alloying Additions, Mater Sci Forum 519-521 (2006) 315-320; F Viana, A M P Pinto, H M C Santos, A B Lopes, Retrogression and re-ageing of 7075 aluminium alloy: microstructural characterization, J Mater Process Technol 92-93 (1999) 54-59; A F Oliveira, M C de Barros, K R Cardoso, D N Travessa, The effect of RRA on the strength and SCC resistance on AA7050 and AA7150 aluminium alloys, Mater Sci Eng A 379 (2004) 321-326; Y Reda, R Abdel-Karim, I Elmahallawi, Improvements in mechanical and stress corrosion cracking properties in Al-alloy 7075 via retrogression and reaging, Mater Sci Eng A 485 (2008) 468-475; G Peng, K Chen, S Chen, H Fang, Influence of repetitious-RRA treatment on the strength and SCC resistance of Al-Zn-Mg-Cu alloy, Mater Sci Eng A 528 (2011) 4014-4018; T Marlaud, A Deschamps, F Bley, W Lefebvre, B Baroux, Evolution of precipitate microstructures during the retrogression and re-ageing heat treatment of an Al-Zn-Mg-Cu alloy, Acta Mater 58 (2010) 4814-4826].

[0006] Solutionizing of the alloys is a pretreatment for thermal aging. During thermal aging of the alloys, the precipitates

that evolve will impede the dislocation movement in the material and impart strength to the alloys. The precipitates that evolve in the grain boundary of the alloys impart EAC resistance to the alloys. The grain boundary precipitates are mainly Mg (Zn,Al,Cu)₂ complexes which are highly anodic in nature. Since Mg is a very active anodic element, the grain boundary precipitates can undergo selective dissolution in the corrosive environments adversely affecting the grain boundary cohesive strength of the alloys. In the presence of stresses, such dissolution can lead to intergranular brittle fracture of the alloys. The rate of intergranular stress corrosion cracking is higher if such anodic precipitates are located continuously at grain boundary. The various thermal aging treatments known and reported in the prior art as stated above do not impart sufficient strength and EAC resistance to the alloys for high strength applications, especially in the aerospace and armor industries and it is still desirable to have aluminium alloys having improved strength and EAC resistance for such applications.

[0007] Therefore, in spite of the several thermal aging treatments that are known and reported for increasing the properties of aluminium alloys, especially the strength and EAC resistance, the need and scope for new and improved thermal aging methods for increasing the properties of 7xxx series aluminium alloys continues. Because of this, over the years, extensive research and development activities are being carried out to devise and develop methods for further improving the strength and EAC resistance of 7xxx series aluminium alloys.

DESCRIPTION OF THE INVENTION

[0008] According to the invention there is provided a four-step thermal aging method for improving environmentally assisted cracking resistance of 7xxx series aluminium alloys, comprising solutionizing and aging 7xxx series aluminium alloys, wherein the aging is carried out by treating the alloys at a temperature of 120 to 130°C for 0.3 to 0.5h, water quenching the alloys to a temperature of 25 to 27°C and further treating the alloys at a temperature of 80 to 95°C for 100 to 120h and at a temperature of 120 to 130°C for 20 to 24h and at a temperature of 155 to 160°C for 1 to 5h and air cooling the alloys to room temperature, sequentially.

[0009] The four-stage thermal aging of the aluminium alloys employing temperature cycles sequentially as described above according to the invention assist in bringing about novel microstructural changes in the alloys so as to improve the EAC resistance of the alloys. It is quite evident from the experimental studies that follow that according to the method of invention more copper is diffused into the grain boundary of the alloys and the distance between the grain boundary precipitates is increased. The copper enrichment and the discontinuously located grain boundary precipitates produce microstructures that significantly reduce the anodic dissolution of the grain boundary precipitates and increase the EAC resistance of the alloys in corrosive environments (especially in 3.5wt.% NaCl). The aging cycles also aid to nucleate more precipitates at the grain matrix and increase the strength levels of the alloys. The thermally aged aluminium alloys of the invention are ideal for high strength applications, especially in the aerospace and armor industries because of their excellent EAC resistance and higher strength. However, they can be used in any other applications also.

[0010] The following experimental examples are illustrative of the invention but not limitative of the scope thereof: The experiments were conducted on dog-bone shaped tensile specimens of AA 7010, AA 7050 and AA 7085 prepared according to ASTM E8. Prior to thermal aging, the alloy samples were solutionized at 460°C and water quenched in known manner. The thermal aging of the samples was carried out in a laboratory scale oil bath furnace fitted with temperature control of accuracy $\pm 2^\circ\text{C}$. Slow strain rate tests (SSRT) were carried out at a strain rate of 10^{-6} s^{-1} and 10^{-7} s^{-1} . Gauge length of the specimens was continuously exposed to 3.5 wt% NaCl that was freely exposed to air, till failure. Each experiment was at least triplicated.

[0011] The electrical conductivity of the heat treated samples were measured using an EDDY current type conductivity meter. EAC susceptibility of the samples was evaluated based on elongation and ultimate tensile strength (UTS) of the samples measured in air (considered as inert) and in corrosive environment (3.5 wt% NaCl freely exposed to air)..

[0012] Fractured samples from SSRT were ultrasonicated in acetone immediately after the failure and were dried and examined under a scanning electron microscope (SEM) to determine the mode of fracture. Ductility (% elongation) of the samples was calculated by measuring the gauge length of the failed samples manually. Microstructures of the specimens were examined in a field emission transmission electron microscope (FEG TEM) operating at 200 kV.

Example 1

[0013] AA 7010 samples in as-received condition were used in the study. A few of the as-received samples were subjected to T6 (peak aged) and T7451 (over aged) temper treatments by us according to the temperature cycles as given in the following Table 2:

Table 2

Thermal treatment	Temperature cycle
Peak aged (PA)	Aged at 100°C for 8 h and further aged at 120°C for 8 h and air cooled
Over aged (OA)	Aged at 100°C for 8 h and further aged at 120°C for 8 h and at 170°C for 8 h and air cooled

[0014] [M Bobby Kannan, R. Raman, A Mukhopadhyay, V S Raja, Influence of multistep aging on the stress corrosion cracking behavior of aluminum alloy 7010, Corrosion. 59 (2003) 881-889; M Bobby Kannan, V S Raja, Role of coarse intermetallic particles on the environmentally assisted cracking behavior of peak aged and over aged Al-Zn-Mg-Cu-Zr alloy during slow strain rate testing, J. Mater. Sci. 42 (2007) 5458-5464; M Bobby Kannan, V S Raja, Enhancing stress corrosion cracking resistance in Al-Zn-Mg-Cu-Zr alloy through inhibiting recrystallization, Eng. Fract. Mech. 77 (2010) 249-256; M Bobby Kannan, V S Raja, Hydrogen embrittlement susceptibility of over aged 7010 Al-alloy, J Mater Sci 41 (2006) 5495-5499; M B Kannan, V S Raja, Enhancing the Localized Corrosion Resistance of High Strength 7010 Al-Alloy, Adv Mater Res 138 (2010) 1-6; Standard Test Methods for Tension Testing of Metallic Materials, ASTM E8].

[0015] A few of the as-received samples were also subjected to thermal treatment according to the method of the invention as per the temperature cycle shown in the following Table 3:

Table 3

Thermal treatment	Temperature cycle
Method of the invention	Aged at 130°C for 0.5h, water quenched to 27°C and further aged at 85°C for 120 h, at 130°C for 24h and at 157°C for 2.5 h

[0016] The SSRT results of the treated AA7010 samples in air (considered as inert) and 3.5 wt% NaCl at strain rate of 10^{-6} s^{-1} were as shown in the following Table 4:

Table 4

Samples	UTS (MPa)	Elongation (%)
Peak aged samples in air	577 (± 4)	13.7 (± 1)
Peak aged samples in 3.5 %wt NaCl	548 (± 3)	7.5 (± 0.8)
Over aged samples in air	539 (± 4)	13.6 (± 1.2)
Over aged samples in 3.5 %wt NaCl	530 (± 3)	12 (± 0.9)
Samples treated as per the invention in air	580 (± 5)	15 (± 2)
Samples treated as per the invention in 3.5 %wt NaCl	569 (± 2)	14 (± 1)

[0017] Table 4 shows that the peak aged samples showed an elongation of about 7.5% in 3.5 wt% NaCl against about 13.7% in air. The drop of elongation in NaCl was about 6.2%. The over aged samples showed an elongation of about 12% in 3.5 wt% NaCl against about 13.6% in air. The drop of elongation in NaCl was about 1.6% compared to air. The samples treated according to the invention showed an elongation of about 14% in 3.5 wt% NaCl against about 15% in air. The drop of elongation in NaCl was only about 1% compared to air. The peak aged samples reached a UTS (ultimate tensile strength) value of about 577 MPa in air compared to about 548 MPa in 3.5wt% NaCl. The drop in strength in 3.5wt% NaCl was about 69 MPa. The over aged samples reached a UTS value of about 539 MPa in air compared to about 530 MPa in 3.5wt% NaCl. The drop in strength in 3.5wt% NaCl was about 9 MPa. The samples according to the invention showed an increased UTS value upto about 580MPa in air and upto about 569 MPa in NaCl. The drop of UTS value in NaCl was about 11 MPa

[0018] The increase in the elongation and UTS values of the samples treated according to the invention are significant and substantial in the overall structural strength and EAC resistance of the 7xxx series aluminium alloys in 3.5 wt% NaCl. The increase or improvement in the elongation and UTS values are indicative of the changes in the microstructures of the samples treated according to the invention. The graph in Fig 1 of the drawings is a representation of stress against % elongation of the alloys in 3.5 wt% NaCl. The graph in Fig 1 supports the findings in Table 4.

[0019] The electrical conductivity values of the heat treated samples were as mentioned in the following Table 5.

Table 5

Samples	%IACS
Peak aged (PA) samples	30
Over aged (OA) samples	36
Samples treated as per the invention	32

[0020] Table 5 shows that the alloy samples treated according to the invention had a lower electrical conductivity as compared to OA samples but had improved EAC resistance as shown by Table 4 above.

[0021] Fractographs or fracture surface maps taken at the edges of the samples treated as per PA and OA temper conditions and according to the invention and failed in 3.5 wt% NaCl were as shown in Figs 2a, 2b and 2c of the drawings, respectively. The peak aged samples of AA7010 in Fig 2a shows extensive intergranular cleavage fracture having brittle facets indicative of stress corrosion cracking (SCC) failure. Table 4 supports this finding in terms of drop in ductility (% elongation). The over aged samples in Fig 2b and the samples according to the invention in Fig 2c did not show any signs of intergranular SCC failure. However, as evident from Table 4, the samples treated according to the invention showed enhanced elongation and strength values even under 3.5 wt.% NaCl clearly indicating that the samples treated according to the invention had better EAC resistance.

[0022] TEM micrographs of the AA 7010 samples peak aged, over aged and treated according to the invention were taken in a FEG TEM and were as shown in Figs 3a, 3b and 3c of the drawings, respectively. The peak aged samples of Fig 3a show finer matrix precipitates and continuous network of fine grain boundary precipitates, whereas the over aged samples of Fig 3b show coarse matrix precipitates and relatively coarser and discontinuous network of grain boundary precipitates. The samples of Fig 3c treated according to the invention show that the grain matrix precipitates remained finer whilst the grain boundary precipitates became coarser thereby improving the strength of the samples. These micrographs clearly establish that the thermal treatment according to the invention brought about significant changes in the microstructure of the alloys.

[0023] Line scan of all the alloy samples were carried out in a FEG TEM in the STEM (Scanning Transmission Electron Microscopy) mode of the FEGTEM using energy dispersive spectroscopy (EDS). A graphical representation of the results is shown in Fig 4 of the drawings. The grain boundary precipitates of the peak and the over aged alloys showed 1.7 and 2.14 wt% Cu respectively. The samples treated according to the invention showed grain boundary precipitates with Cu enrichment of 5.9 wt%. Cu enrichment in the grain boundary precipitates of the samples treated according to the invention is understood to be the key factor for the enhanced EAC resistance of the samples as Cu in the precipitates is known to increase the EAC resistance of the alloys. The higher volume fraction of matrix precipitates shown in the TEM micrographs is believed to attribute to the increased strength of the alloys.

Example 2

[0024] AA 7050 samples in as-received condition were used for the study. The samples as-received were already treated according to T7451 (OA temper condition). A few of the samples were further subjected to thermal aging according to the invention under the same conditions as given in Table 3 of Example 1. The SSRT results for the AA 7050 samples as-received and after the thermal treatment according to the invention in air and 3.5 wt% NaCl were as shown in the Table 6 below:

Table 6

Samples	Condition	UTS (in MPa)	% Elongation
As-received and over aged	Air	523	15.3 ± 0.9
	NaCl (10 ⁻	511	13.9 ± 0.4
	NaCl (10 ⁻	478	11.01 ± 0.4
Treated as per the invention	Air	600	16.4 ± 1.6
	NaCl (10 ⁻	597	15.2 ± 1.1
	NaCl (10 ⁻⁷ /s	543	13.56 ± 0.4

[0025] The as-received and over aged samples showed an elongation of about 13.92 % in 3.5 wt% NaCl against about 15.25 % elongation in air. The as-received and over aged samples had a UTS value of about 523 MPa in air compared

to about 511 MPa in 3.5 wt% NaCl. The samples treated as per the invention showed about 16.36% elongation in air and about 15.18% elongation in 3.5 wt% NaCl. The samples treated as per the invention showed a UTS value of about 600 MPa in air and about 597 MPa in NaCl. The samples treated according to the invention exhibited significant improvement in the strength and EAC resistance as compared to the over aged samples even at a strain rate of 10^{-7} /s. The graphical representation of stress vs % elongation in NaCl of the as-received and over aged samples and samples treated according to the invention as shown in Figs 5a and Fig 5b of the drawings, respectively supports the finding in Table 6.

[0026] The electrical conductivity values of the treated samples were as mentioned in the following Table 7.

Table 7

Samples	%IACS
Over aged samples	39
Samples treated as per the invention	33

[0027] Table 7 shows that the alloy samples treated according to the invention had a lower electrical conductivity as compared to OA samples but had improved EAC resistance as shown by Table 6 above.

[0028] Fractographs on the edges of the as-received and over aged samples and samples treated according to the invention that failed in 3.5 wt% NaCl were taken in a SEM and were as shown in Figs 6a and 6b of the drawings respectively. The as-received and over aged samples of Fig 6a show indications of extensive shear overload failure whereas the treated samples of Fig 6b indicate a microvoid coalescence failure. The microvoid coalescence of the treated samples in Fig 6b is indicative of delayed time-to-failure as evidenced by the numerous dimples in the microstructure. The elongation values in Table 6 support this finding.

[0029] TEM micrographs of the as-received and over aged samples and samples treated according to the invention were taken in a FEG TEM and were as shown in Figs 7a and 7b of the drawings respectively. The as-received and over aged samples of Fig 7a show a discontinuous network of grain boundary precipitates, whereas the treated samples of Fig 7b show a relatively coarser and more discontinuous grain boundary precipitates. A graphical representation of % Cu in the grain boundary precipitates of the samples taken through FEG TEM is shown in Fig 8 of the drawings. The grain boundary precipitates of the over aged alloys and alloys treated as per the invention showed 3.4 and 5.3 wt% Cu respectively. The microstructural changes in the grain boundary precipitates are understood to be the reasons or causative factors for the improved EAC resistance of the treated samples.

Example 3

[0030] AA 7085 samples in as-received condition were used for the study. The samples as-received were already thermally treated according to T7651 (OA temper condition). A few of the samples were further treated according to the invention under the same conditions as given in Table 3 of Example 1. The SSRT results for the AA 7085 samples in as-received and over aged condition and after the thermal treatment according to the invention in 3.5 wt% NaCl were as shown in Table 8 below.

Table 8

Samples	3.5 wt% NaCl	UTS (in MPa)	% Elongation
As-received and over aged	10^{-6} s^{-1}	500	5.5 ± 0.2
	10^{-7} s^{-1}	485	3.3 ± 0.4
Treated as per invention	10^{-6} s^{-1}	520	8 ± 0.8
	10^{-7} s^{-1}	498	7 ± 0.6

[0031] The as-received and over aged samples showed an elongation of about 5.5 % at a strain rate of 10^{-6} s^{-1} , whereas the elongation dropped to about 3.3 % at a strain rate of 10^{-7} s^{-1} in 3.5 wt% NaCl. The samples treated as per the invention showed high strain to failure at the strain rate of 10^{-6} s^{-1} and even at the strain rate of 10^{-7} s^{-1} in 3.5 wt. % NaCl indicating that the samples treated as per the invention had significantly increased the EAC resistance. The samples treated as per the invention also had significantly higher strength (MPa) values. The graphical representation of stress vs % elongation of the samples in 3.5 wt. % NaCl as shown in Fig 9 of the drawings supports the finding in Table 8.

[0032] Fractographs of the edges of the as-received and over aged samples and samples treated according to the

invention that failed in 3.5 wt.% NaCl were taken in a SEM and were as shown in Figs 10a and 10b of the drawings, respectively. The as-received and over aged samples in Fig 10a show indications of extensive shear overload failure whereas samples treated as per the invention in Fig 10b indicate a microvoid coalescence failure. The microvoid coalescence of the samples treated as per the invention in Fig 10b is indicative of delayed time-to-failure as evidenced by the numerous dimples in the microstructures. The elongation values in Table 6 support this finding.

[0033] TEM micrographs of the as-received and over aged samples and samples treated according to the invention were taken in a FEGTEM and the images were as shown in Figs 11a and 11b of the drawings respectively. The as-received and over aged samples in Fig 11a show a discontinuous network of grain boundary precipitates, whereas the samples treated as per the invention in Fig 11b show a relatively coarser and more discontinuous grain boundary precipitates. The microstructural changes in the grain boundary precipitates are understood to be the reasons or causative factors for the improved EAC resistance of the treated samples as per invention.

[0034] It is quite evident from the experimental studies that the 7xxx aluminium alloy samples treated according to the invention showed significant and substantial changes in the microstructures of the alloys. Enrichment of Cu in the grain boundary precipitates is understood to raise the galvanic potential of the alloys and resist their tendency for preferential dissolution in corrosive environment and as a consequence increase their EAC resistance significantly. It is reported that suppression of preferential dissolution of grain boundary precipitates can lead to better EAC resistance. [R Goswami, S Lynch, N J H Holroyd, S P Knight, R L Holtz, Evolution of grain boundary precipitates in Al 7075 upon aging and correlation with stress corrosion cracking behavior, Metall. Mater Trans A Phys Metall Mater Sci 44 (2013) 1268-1278; S P Knight, N Birbilis, B C Muddle, A R Trueman, S P Lynch, Correlations between intergranular stress corrosion cracking, grain-boundary microchemistry, and grain-boundary electrochemistry for Al-Zn-Mg-Cu alloys, Corros. Sci. 52 (2010) 4073-4080; S P Knight, K Pohl, N J H Holroyd, N Birbilis, P A Rometsch, B C Muddle, et al, Some effects of alloy composition on stress corrosion cracking in Al-Zn-Mg-Cu alloys, Corros. Sci. 98 (2015) 50-62; M Puiggali, A. Zielinski, J M Olive, E Renauld, D Desjardins, M Cid, Effect of microstructure on stress corrosion cracking of an Al-Zn-Mg-Cu alloy, Corros. Sci 40 (1998) 805-819].

[0035] It is evident from Figs 3b vs Figs 3c that the size of the grain boundary precipitates is larger for the samples treated according to the invention. The larger grain boundary precipitates appear to act as trapping sites for atomic hydrogen and thereby reduce hydrogen concentration at the crack tip of the alloys and result in improved EAC resistance. [G M Scamans, Hydrogen bubbles in embrittled Al-Zn-Mg alloys, J Mater Sci 13 (1978) 27-36; K Rajan, W Wallace, J C Beddoes, Microstructural study of a high-strength stress-corrosion resistant 7075 aluminium alloy, J Mater Sci 17 (1982) 2817-2824]. The SSRT data and the fractographs showing predominant dimple regions in all the samples treated as per the invention indicate high EAC resistance without sacrificing or comprises the strength levels.

[0036] Although it was known and reported that the composition and morphology of the grain boundary precipitates of aluminium alloys largely determine the EAC resistance of the alloys and that Cu enrichment of the alloys in the grain boundary can bring changes in the microstructures and thereby increase the EAC resistance of the alloys, the prior art does not teach or teach towards the four-step aging cycle and the temperatures and aging durations and the sequence of lowering and raising the temperatures of method of the invention that brings about the changes in the microstructure of the alloys. Therefore, the method of the invention is unique, innovative and inventive. The method of the invention has been found to be effective in 7xxx aluminium alloys having a wide range of quench sensitivity. Therefore, it is believed that the method of the invention is applicable to at least all the 7000 series aluminium alloys, if not aluminium alloys in general.

Claims

1. A four-step thermal aging method for improving environmentally assisted cracking resistance of 7xxx series aluminium alloys, comprising solutionizing and aging 7xxx series aluminium alloys, wherein the aging is carried out by treating the alloys at a temperature of 120 to 130°C for 0.3 to 0.5h, water quenching the alloys to a temperature of 25 to 27°C and further treating the alloys at a temperature of 80 to 95°C for 100 to 120h and at a temperature of 120 to 130 °C for 20 to 24h and at a temperature of 155 to 160°C for 1 to 5h and air cooling the alloys to room temperature, sequentially.
2. The method as claimed in claim 1, wherein the alloys are treated at a temperature of 130°C for 0.5 h, water quenched to 27°C and further treated at a temperature of 85°C for 120h, at a temperature of 130°C for 24h, and at a temperature of 157°C for 2.5h.
3. The method as claimed in claim 1 or 2, wherein the alloys are AA 7010, AA 7050, and AA 7085.

Patentansprüche

1. Vierstufiges Verfahren einer thermischen Alterung zur Verbesserung der Beständigkeit von Aluminiumlegierungen der 7xxx-Serie gegen umweltbedingte Rissgefahr ("environmentally assisted cracking" (EAC)), umfassend ein Lösungsglühen und Altern der Aluminiumlegierungen der 7xxx-Serie, wobei das Altern ausgeführt wird, indem die Legierungen für 0,3 bis 0,5 h bei einer Temperatur von 120 bis 130 °C behandelt werden, Wasserabschrecken der Legierungen auf eine Temperatur von 25 bis 27 °C und Weiterbehandeln der Legierungen, schrittweise nacheinander, für 100 bis 120 h bei einer Temperatur von 80 bis 95 °C und für 20 bis 24 h bei einer Temperatur von 120 bis 130 °C und für 1 bis 5 h bei einer Temperatur von 155 bis 160 °C und Luftkühlen der Legierungen auf Raumtemperatur.
2. Verfahren nach Anspruch 1, wobei die Legierungen für 0,5 h bei einer Temperatur von 130 °C behandelt werden, auf 27 °C wasserabgeschreckt und für 120 h bei einer Temperatur von 85 °C, für 24 h bei einer Temperatur von 130 °C und für 2,5 h bei einer Temperatur von 157 °C weiterbehandelt werden.
3. Verfahren nach Anspruch 1 oder 2, wobei die Legierungen AA 7010, AA 7050 und AA 7085 sind.

Revendications

1. Procédé de vieillissement thermique en quatre étapes pour l'amélioration de la résistance à la fissuration favorisée par l'environnement d'alliages d'aluminium de la série 7xxx, comprenant un traitement thermique en solution et le vieillissement d'alliages d'aluminium de la série 7xxx, dans lequel le vieillissement est effectué par traitement des alliages à une température de 120 à 130 °C pendant 0,3 à 0,5 h, trempe à l'eau des alliages jusqu'à une température de 25 à 27 °C et traitement encore des alliages à une température de 80 à 95 °C pendant 100 à 120 h et à une température de 120 à 130 °C pendant 20 à 24 h et à une température de 155 à 160 °C pendant 1 à 5 h et refroidissement à l'air des alliages jusqu'à la température ambiante, successivement.
2. Procédé tel que revendiqué dans la revendication 1, dans lequel les alliages sont traités à une température de 130 °C pendant 0,5 h, trempés à l'eau jusqu'à 27 °C et encore traités à une température de 85 °C pendant 120 h, à une température de 130 °C pendant 24 h et à une température de 157 °C pendant 2,5 h.
3. Procédé tel que revendiqué dans la revendication 1 ou 2, dans lequel les alliages sont AA 7010, AA 7050 et AA 7085.

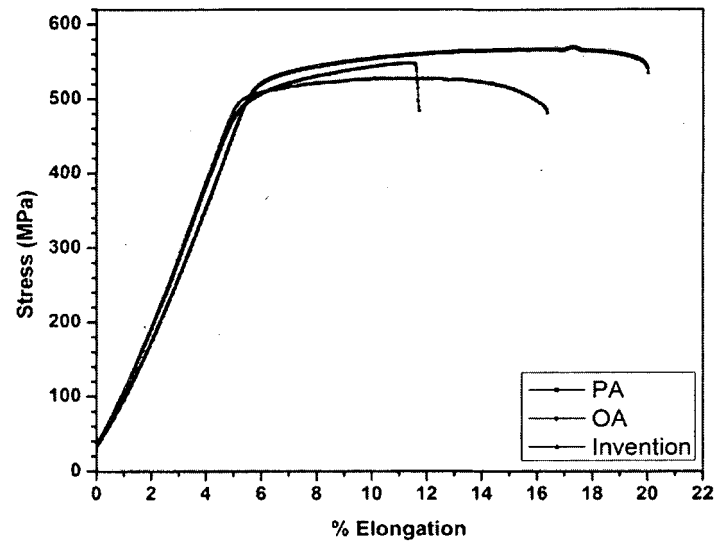


Fig 1

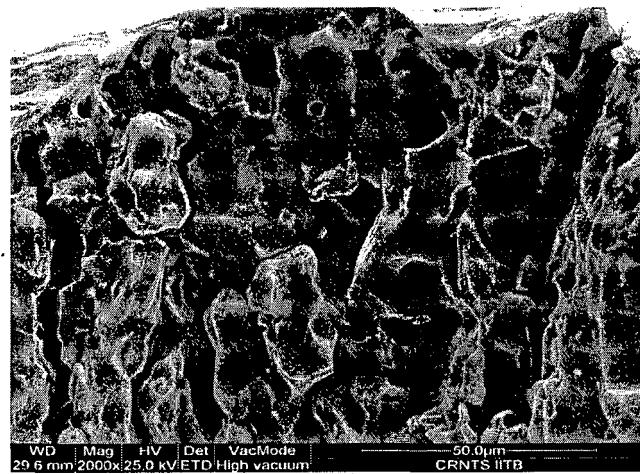


Fig 2a

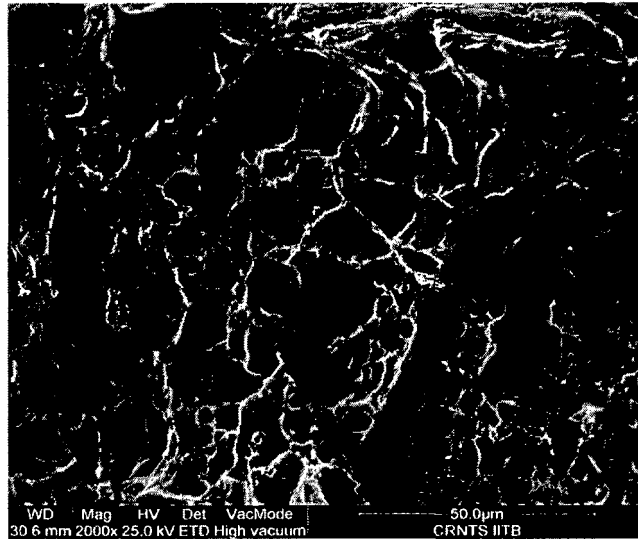


Fig 2b

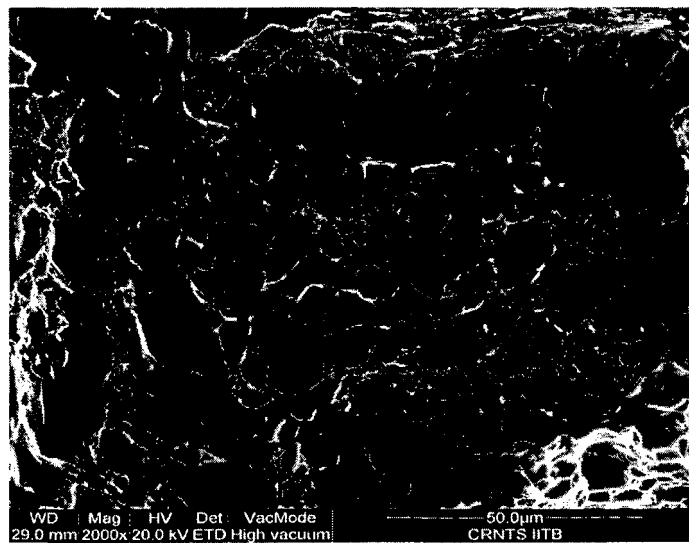


Fig 2c

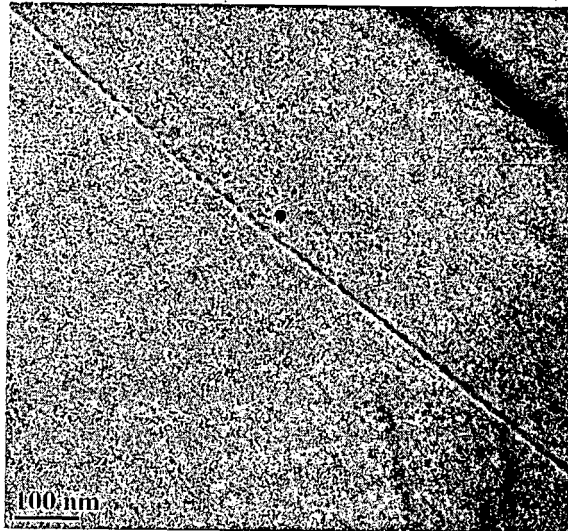


Fig 3a

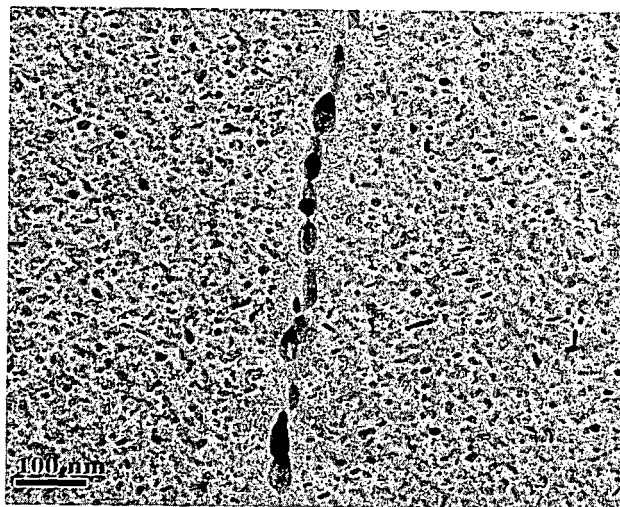


Fig 3b

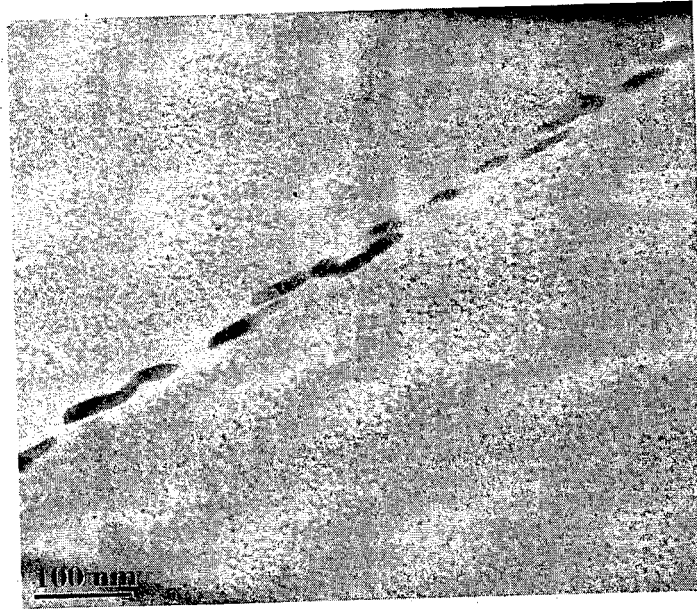


Fig 3c

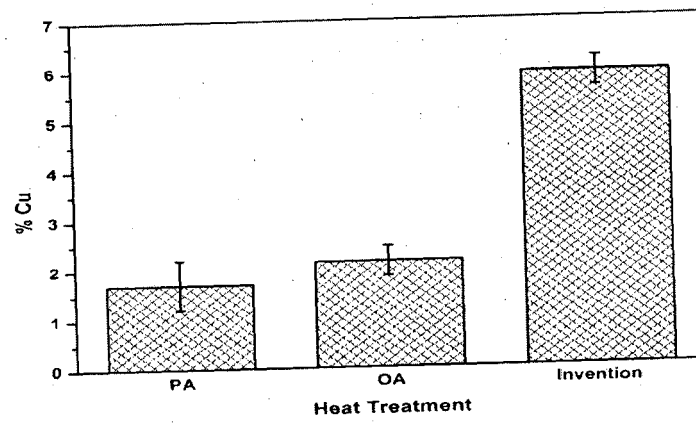


Fig 4

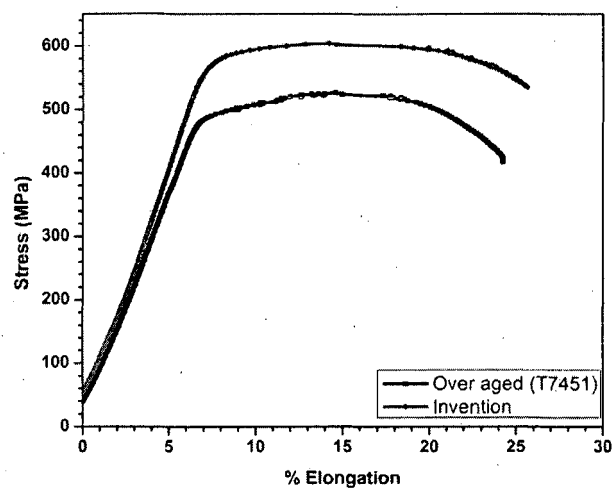


Fig 5a

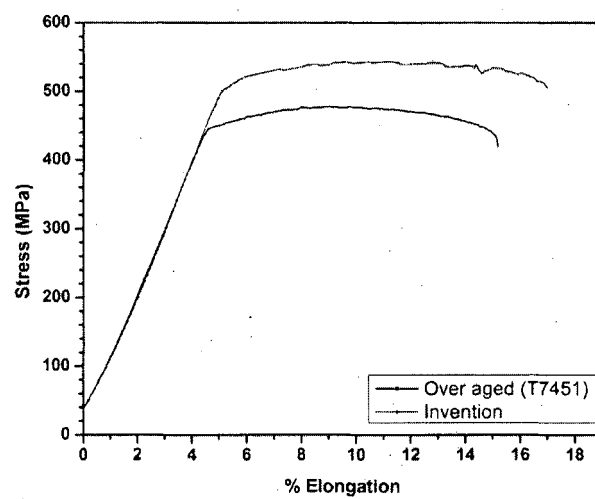


Fig 5b



Fig 6a

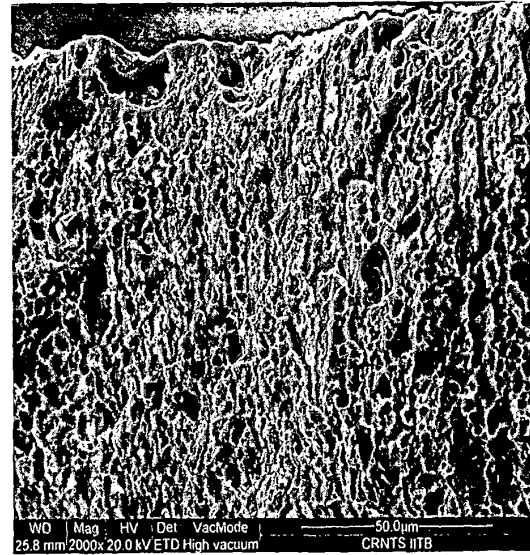


Fig 6b

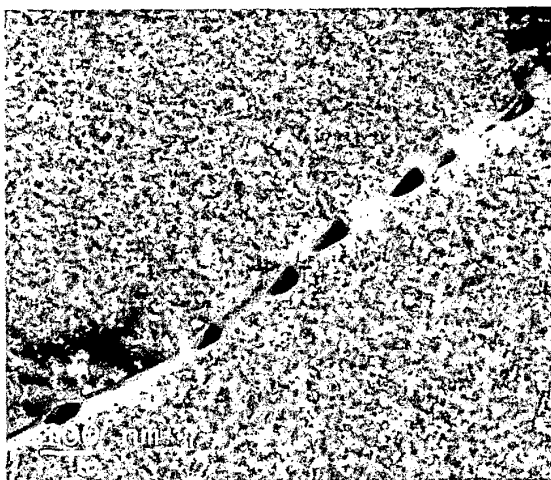


Fig 7a

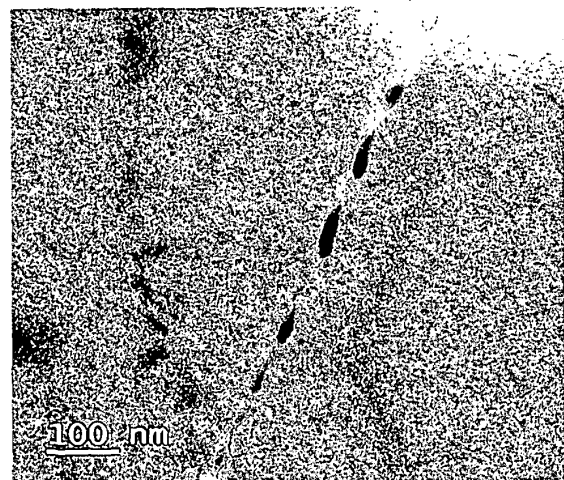


Fig 7b

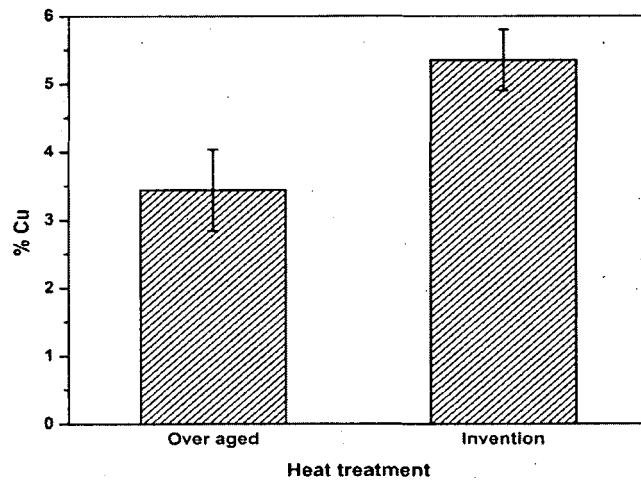


Fig 8

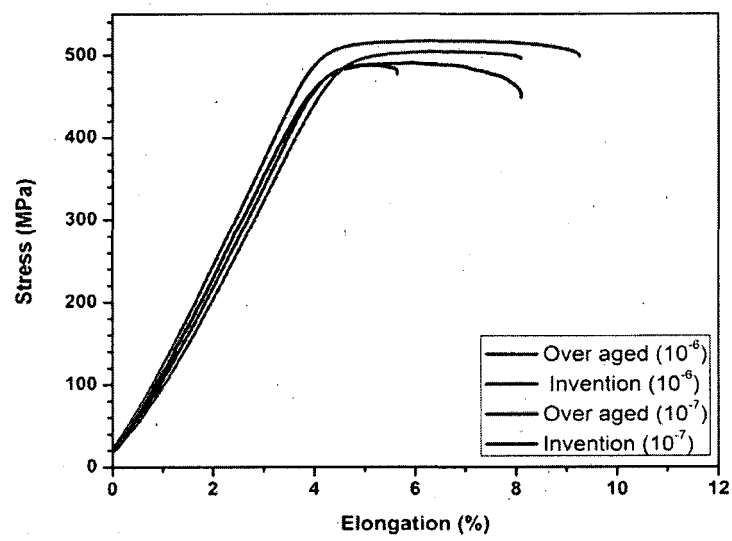


Fig 9

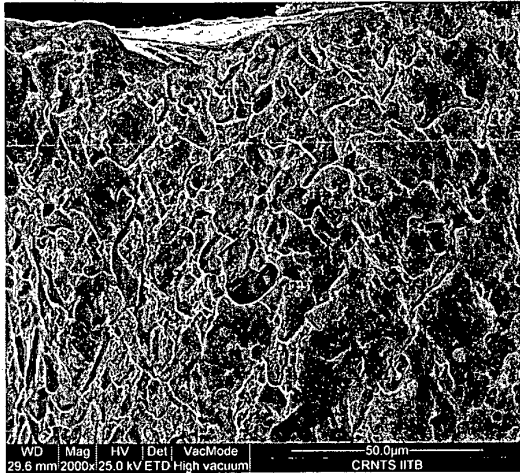


Fig 10a

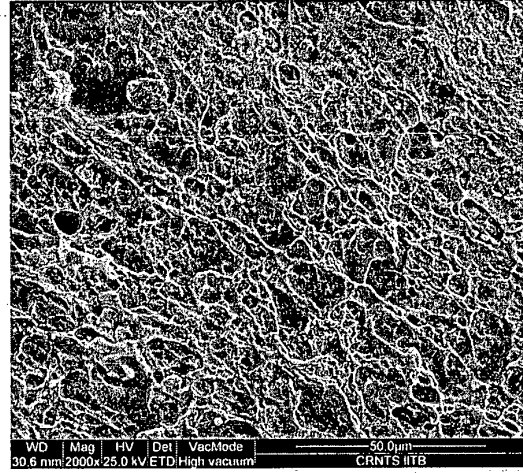


Fig 10b

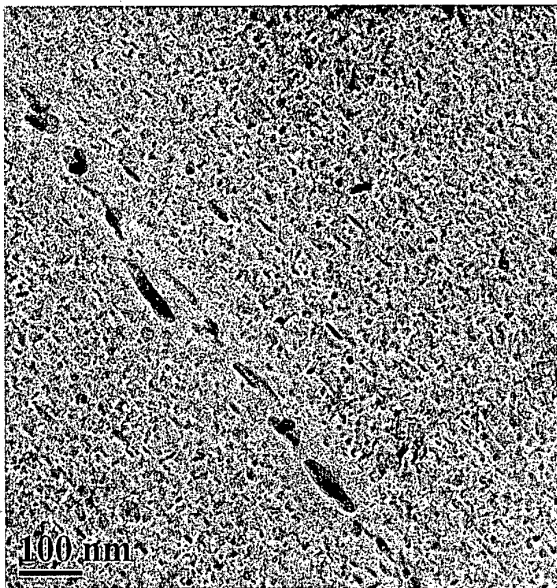


Fig 11a

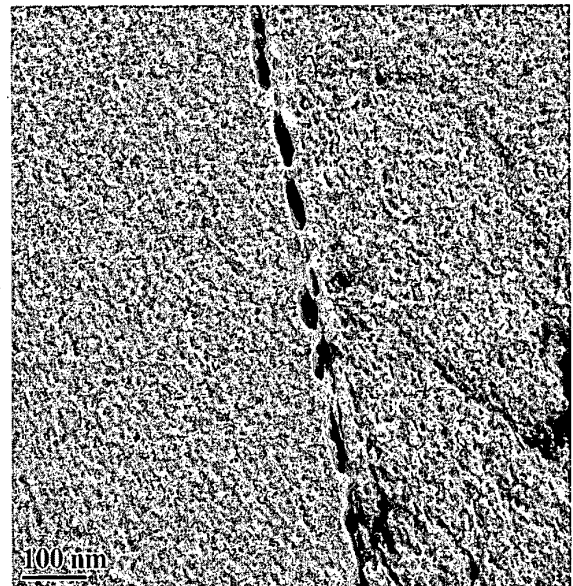


Fig 11b

REFERENCES CITED IN THE DESCRIPTION

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