Ratko Mimica, ME

Assistant Department of Manufactural Engineering Faculty of Electrical Engineering, Mechanical Engineering and Naval Arhitecture University of Split E-mail: rmimica@fesb.hr

HARDNESS VERSUS TIME DEPENDENCY DURING ARTIFICIAL AGEING OF AlMgSi0.5 ALUMINIUM ALLOY

UDK / UDC: 669.715 JEL klasifikacija / JEL classification: L51 Stručni rad / Professional paper Primljeno / Received: 2. lipnja 2015. / June 2, 2015 Prihvaćeno za tisak / Accepted for publishing: 17. studenog 2015. / November 17, 2015

Abstract

Al-Mg-Si aluminium alloy are characterized by excellent deformability, but mechanical properties are not significant in extruded state. Improvement of mechanical properties is achieved by heat treatment, a process which allows formation of metastable precipitates during subsequent ageing. In this work, hardness versus time dependency for artificially aged AlMgSi0.5 (EN AW-6060) aluminium alloy at 185°C is presented, along with qualitative and quantitative analysis of results.

Key words: AlMgSi0.5 aluminium alloy, heat treatments, metastable precipitates

1. INTRODUCTION

Aluminium is increasingly attractive due to its strength and stiffness to weight ratio. Al-Mg-Si alloys are age hardenable and widely used in both cast and wrought forms. Aluminium extrusions are used in a variety of structural applications ranging from building and automotive to aerospace industries. Al-Mg-Si series alloys claim over a 90% share of the total extruded volume owing to an attractive combination of mechanical properties, corrosion resistance, extrudability and an excellent response to surface finishing operations (Birol 2013; Meng 2010). In extruded state mechanical properties are not significant so strengthening of Al-Mg-Si alloys is based on a precipitation hardening process. This series of alloys are available in different ratios of Mg and Si, so that different properties could be optimized. Al-Mg-Si alloys are considered corrosion resistant, especially in atmospheric conditions, while in presence of chloride media breach of passive film was observed which suggests pitting corrosion susceptibility (Mimica, Radošević & Matešić 2011).

Magnesium and silicon are the main alloying elements and combine to form the stoichiometric compound, magnesium silicide (Mg_2Si) when the weight ratio of magnesium to silicon is 1.73:1. The Mg_Si makes Al-Mg-Si series alloys heat treatable (Meng 2010). Heat treatment provides a way to change microstructure by modifying size, spacing, composition, type and precipitate morphology in heat treatable aluminium alloy.

Heat treatment usually comprises 3 main stages, namely:

- Solution heat treatment at a higher temperature within the single phase region, so as to dissolve the alloying elements;
- Quench, in order to form a supersaturated solid solution (SSSS) of these elements in Al, and
- Ageing, i.e. the controlled decomposition of the supersaturated solid solution to form a fine dispersion of precipitates. This can occur at any temperature below the solvus line, even at room temperature.

Temperatures and time kept on these temperatures are chosen in dependence of chemical composition of the alloy or desired structure to be formed. Upon ageing, a supersaturated solid solution will tend to transform towards the equilibrium structure $\beta - Mg_2Si$. However, before equilibrium is attained, a number of intermediate stages typically occur as can be seen in table 1.

Table 1.

Stage	Solute concentration (at.%)	Shape	Description, features		
SSSS	0-5%*	Point defects	Supersaturated solid solution: substitutional Mg and Si atoms, large concentration of vacancies.		
Atomic clusters	1-15%*	clusters	Early clustering of solute atoms, large degeneracy in formation enthalpy of structures, entropy effects very strong.		
Clusters (1-5 nm)	10-25%	3d, 2d	Spherical and platelets		
initial-β"	15-45%	needle	Monoclinic cell, single Si pillars		
pre-β"	35-60%	needle	Monoclinic cell, double Si pillars, low density cylinder appears(LDC)		
β"	50-90%	needle	Monoclinic cell, double Si pillars, LDC underwent 0.5b shift		
Β', β', U1, U2, U3	60-100%	rod/lath	Collection of hexagonal, trigonal and orthorhombic phases		
β	95-100%	cube	Stable Mg ₂ Si bulk phase with anti-fluorite structure, excess Si forms the diamond structure		

Overview of the complex precipitation sequence in Al-Mg-Si alloys (Huis 2007)

* No real distinction can be made between the precipitate and the matrix. The particles are too immature and contain too much Al to determine where the interface is.

Precipitation hardening effect depends on the shape, size and morphology of the precipitates formed during ageing. Four main strengthening mechanisms are explained in Fig 1.



- **1.** Solute hardening effect diminishes with time because precipitate formation leads to reduction of solute in the α phase.
- **2.** Coherency strain hardening. In the beginning, precipitates are too finely spaced for dislocations to bend around them; in later stages spaces are too wide. Maximum coherency strain hardening occurs when the average zone spacing is similar to the limiting radius of dislocation curve.
- **3.** Chemical hardening is the increase in stress required to force a dislocation through a coherent zone/precipitate
- **4. Dispersion Hardening**. At higher ageing times and/or temperatures, precipitates become incoherent and dislocations are no longer able to cut through them.



It's important to note that these mechanisms are not mutually exclusive - many alloys are designed to take advantage of all four (aluMATTER 2010).

There are numerous temper designations that are standardized for heat treated aluminum alloys (Benedyk 2009), in addition, newly developed multistage thermal treatments have shown significant improvement in mechanical properties(Lumley & Morton 2004). Al-Mg-Si series alloys are usually delivered in naturally aged (T4) or artificially aged (T6) temper condition, without any insight in precipitation dynamic which preceded final structure. The scope of this work is to obtain hardness vs. ageing time dependency for chosen alloy, which will be used as baseline in further research related to multistage thermal treatments.

2. EXPERIMENTAL PROCEDURE

Experimental part of this work was conducted in welding laboratory of Department of Manufactural Engineering in Faculty of Electrical Engineering, Mechanical Engineering and Naval Architecture in Split. Material used is AlMgSi0,5 (EN-AW 6060) aluminum alloy with chemical composition shown in table 2.

Table 2.

Mg	Si	Cu	Fe	Mn	Cr	Ti	Zn	Al
0,44	0,44	0,01	0,19	0,06	0,01	0,02	0,02	rest

Chemical composition AlMgSi0,5 alloy used in this investigation (%)

Base material is purchased in extruded form of a strip dimensions 40 x 5 x 6000[mm], temper designation was T6 which implies artificial ageing to peak hardness. Samples dimensions 5x8x40[mm] were cut from base material, and afterwards grinded using 240, 360, 480 i 1000 grit sandpaper in order to minimize hardness measurements dispersion. The hardness test, because of its simplicity and correlation with tensile properties is a common mechanical test used to estimate the mechanical strength of metallic materials(Malin 1995; Meng 2010). Vickers hardness measurements (2 kg load) were performed for each sample. Values presented are average of five measurements. All measurements are conducted in compliance with ASTM E18-07 standard.

Heat treatment of samples included solid solution heat treatment (SSHT), quenching and artificial ageing. SSHT was carried out at 540°C for period of 1 hour. Quenching must be performed fast enough to avoid diffusion processes and loss of vacancies, easiest way to do that is quench in water at ambient temperature. Artificial ageing was performed at 185°C as a compromise between overall ageing time and required mechanical properties.

3. **RESULTS AND DISCUSSION**

Base material hardness in as received (T6) condition is 80 HV2. Hardness versus ageing time curve for samples after SSHT and quenching is shown in Fig 2. Curve shape resembles typical two stage hardening curve which is common for heat treatable aluminium alloys. Base material hardness was achieved and surpassed in relatively wide time interval (9 to 15 hours of artificial ageing). Reasons for such behavior could be associated with different heat transfer conditions in manufacturing process compared to samples heated in laboratory (dimensions, heating and cooling rates, etc.). Also, extended area of increased hardness has positive effect on robustness of the manufacturing process, making it easier to achieve values set by temper standards.



Figure 2. Hardness vs. ageing time at 185°C for AlMgSi0.5 aluminium alloy

Fig 2. also shows dispersion of the results which usually follows hardness measurements. Namely, besides magnesium and silicon, other alloying elements and impurities are also dissolved in aluminium matrix, so inhomogeneity of structure and thus the measurements is to be expected. But despite dispersion, average values clearly outline changes in hardness related to artificial ageing time. Hardness gradient is positive during first stages of ageing, with approximate value of 5 HV/hour (Vickers per hour). After 5 hours of ageing gradient gradually decreases and finally comes to zero (no further rise in hardness) at 9th hour of artificial ageing. In ageing time interval between 9 and 15 hours overall hardness firstly decreases (negative hardness gradient) and then again increases to maximum averaged value of 87 HV2. After that period hardness decreases at approximately constant rate of 1,5 HV/hour.

Comparing the measured average values with Fig 1. could provide insight in interaction of strengthening mechanisms active during artificial ageing (Fig 3.).

Ideal fit between curves is not to be expected, firstly, because of abovementioned inhomogeneity, and secondly because first figure is related to increase in tensile strength which may not be in linear correlation with hardness. But nevertheless, understanding the resemblance between these two curves will be beneficial for further investigations of multistage thermal treatments.



Figure 3. Schematic interpretation of results based on hardening mechanisms synergic effect.

Solution hardening in this alloy could not be dominant process mainly because AlMgSi0,5 have smallest amount of alloying elements compared to other alloys from that series. During artifitial ageing, nucleation and growth of the precipitates further depletes the aluminium matrix from solute, reducing contribution to overall hardness from this mechanism.

Rapid increase in hardness during first hours of ageing may be explained with coherency strain hardening mechanism. Previous research (Huis 2007) related to early stages of precipitations in Al-Mg-Si alloys have shown that early phases (clusters, initial- β ", pre- β " and β ") maintain coherency with aluminium matrix. And as a result of different chemical composition formation of strain fields which impede dislocation motion is to be expected. At first five hours of ageing precipitates are too finely spaced for the dislocation to bend around strain fields. Dislocation therefore threads trough strain fields which has limited increase in dislocation obstruction and therefore in overall hardening effect. At later stages (from 10 hours of artificial ageing onwards) precipitates are too widely spaced, so dislocation can glide between them relatively unhindered. Maximum coherency strain hardening (recorded just before first hardness peak at 9-th hour of ageing) occurs when the average zone spacing is similar to the limiting radius of dislocation curvature. During later stages of ageing precipitation of partially incoherent phases such as β ' occurs. New dislocations are formed on particle/matrix interface which reduces hardening effect by this mechanism, and with formation of incoherent (stable) phases coherency strain hardening effect ends.

Secondary peak in hardness (secondary hardening), observed after 13th hour of ageing and subsequent decrease after 15th hour could be result of two interactive mechanisms, chemical hardening and dispersion hardening. The increase in stress required to force a dislocation through a coherent or partially incoherent precipitate is termed chemical hardening. Hardening effect increases with radius of precipitate until moment at which less energy is required for dislocation to bow around precipitate instead of cutting through. In literature, this effect is known as Orowan bowing, and that radius of a particle is called critical radius. From that point onwards i.e., for particle radius larger than critical, dislocation must by-pass precipitate by one of a number of possible mechanisms (bowing, climb, cross-slip) making dispersion hardening the dominant mechanism. At longer ageing times, precipitates start to coarsen and the strengthening effect decreases.

It is interesting to notice that coherency strain hardening in this type of alloy offers almost same amount as combined effect of next two mechanisms (chemical and dispersion hardening), i.e., effect of secondary hardening is not pronounced like in Al-Cu or Al-Zn series of aluminium alloys (Lumley & Morton 2004). In those types of alloys secondary hardening is interpreted with different sequence, shape and properties of metastable precipitates, although partially incoherent, still offer significant contribution to hardening. But, even in same series of alloys like AlMgSi0,8 or AlSi1MgMn effect of secondary hardening is noticeable (Chen 2006). Reasons for that kind of behavior could be related to the small amount of alloying elements contained in AlMgSi0,5 alloy. During first stages of ageing large number of small precipitates are formed impeding dislocation motions with strain fields induced in aluminium matrix. Those strain fields are not solely dependent on particle radius, atom size or lattice mismatch of a formed precipitate in respect to matrix also make contribution to overall hardening effect. Particle radius plays much more important role in chemical and dispersion hardening mechanism, as mentioned above. So, smaller amount of alloying elements (building blocks of future precipitates) would definitely lead to smaller number of precipitates with critical radius size, and thus smaller hardening effect.

4. CONCLUSION

Understanding the precipitation dynamic and hardening mechanisms is essential for optimization of heat treatments and mechanical properties of

heat treatable aluminium alloys. In this work, change in hardness related to ageing time at 185°C was investigated for AlMgSi0,5 alloy. Hardness vs. time curves shows two peaks, first after 9 hours of ageing and second after 15 hours. Maximum hardness is 87HV2 recorded during 15th hour of ageing. These results will be used as baseline in further research of multistage thermal treatments Al-Mg-Si aluminium alloys.

REFERENCES

Birol, Y., 2013. Precipitation during homogenization cooling in AlMgSi alloys. *Transactions of Nonferrous Metals Society of China (English Edition)*, 23(October 2012), pp.1875–1881.

Meng, C., 2010. *Effect of Preheating Condition on Strength of AA6060 Aluminium Alloy for Extrusion*.Ph. D thesis, Auckland University of Technology.

Mimica, R., Radošević, J. & Matešić, S.-S., 2011. Elektrokemijska svojstva aluminijske slitine EN AW 6060. *strojarstvo*, 53(4), pp.199–202.

Van Huis, M. a. et al., 2007. Phase stability and structural features of matrix-embedded hardening precipitates in Al-Mg-Si alloys in the early stages of evolution. *Acta Materialia*, 55, pp.2183–2199.

Precipitation hardening overview 2010. Available from: http://aluminium.matter.org.uk/content/html/eng/default.asp?catid=61&pageid=989848382, [15 March 2015]

Benedyk, J.C., 2009. International Temper Designation Systems for Wrought Aluminum Alloys. *Light Metal Age*, 67, pp.3–6. Available at: https://lightmetalage.com/PDFs/LMA-2009-10-026TempersPartI.pdf.

Lumley, R.N., Polmear, I.J. & Morton, a. J., 2004. Temper developments using secondary ageing. *Materials Forum*, 28, pp.85–95.

Malin, V., 1995. Study of Metallurgical Phenomena in the HAZ of 6061-T6 Aluminum Welded Joints. *Welding journal*, pp.305–318.

Chen, J.H. et al., 2006. Atomic pillar-based nanoprecipitates strengthen AlMgSi alloys. *Science (New York, N.Y.)*, 312(2006), pp.416–419.

Ratko Mimica, dipl. ing.

Asistent Zavod za proizvodno strojarstvo, Split Fakultet elektrotehnike, strojarstva i brodogradnje E-mail: rmimica@fesb.hr

OVISNOST TVRDOĆE O VREMENU UMJETNOG DOZRIJEVANJA ALUMINIJSKE LEGURE AIMgSi0.5

Sažetak

AlMgSi aluminijske legure karakterizira odlična deformabilnost, ali u ekstrudiranom stanju mehanička svojstva nisu značajna. Poboljšanja mehaničkih svojstava postižu se toplinskim očvršćivanjem koje je posljedica formiranja metastabilnih faza tijekom procesa naknadnog dozrijevanja. U ovom je radu istražena krivulja ovisnosti tvrdoće o vremenu umjetnog dozrijevanja na 185 °C za AlMgSi0.5 (EN AW-6060) aluminijsku leguru i izloženo je tumačenje iznosa i oblika istražene krivulje.

Ključne riječi: aluminijska legura AlMgSi0.5, toplinsko očvršćavanje, metastabilni precipitati.

JEL klasifikacija: L51