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Influence of process-induced microstructure on hardness of two Al–Si alloys

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ABSTRACT

This paper analyses the influence of thermal-induced aging on the strength of cast A319 and A356 aluminum alloys. This phenomena is of primary importance especially in the automotive industry, where on one hand the alloys experience Thermo-Mechanical and Low Cycle Fatigue loading conditions and on the other hand their microstructure is induced both by the manufacturing process and the thermal histories. The alloys studied here were produced using an industrial Lost Foam Casting process which affects significantly the microstructure and the precipitation compared to standard Die Casting process. The paper compares mechanical properties of these peculiar alloys in terms of microhardness at the macroscopic level as a function of thermal overaging time and at the microscopic level in terms of properties of the different phases (intermetallics, eutectics) present in the microstructure. Important differences can be observed between these different phases and the results are consistent with similar studies from the literature on different aluminum alloys and processes.

1. Introduction

Thermo-Mechanical Fatigue (TMF) of cylinder head fire decks is nowadays a critical design aspect in the automotive industry as a consequence of new constraints imposed by fuel efficiency, engine performances and polluting emission reduction. From a material point of view, this application experiences operating temperatures up to 250 °C which are very high for the aluminum alloys commonly used for the manufacturing of these parts. The alloy indeed locally reaches 50–70% of its melting temperatures and exceeds the maximal temperatures of the heat treatments that could be used for the stabilization of its microstructure (T7 for instance). Moreover, these operating temperatures induce plasticity, creep and ageing effects which need to be considered in a durability assessment of the structure [1].

Until now, gravity Die Casting (DC) with permanent dies has been the common choice for the manufacturing of the cylinder head. For some years now, the Lost Foam Casting (LFC) process has become a new standard manufacturing process as it enables the realization of intricate parts with an excellent surface finish and an important cost reduction due to the suppression of the expensive metallic molds and the decrease of the energetic costs. The LFC process has however some drawbacks, when compared to DC. It shows a low solidification rate and consequently produces a coarser microstructure expressed in terms of grain size and SDAS (Secondary Dendrite Arm Spacing), with numerous intermetallic and eutectic phases and residual porosity formed during the degradation of the polymeric pattern [2,3].

The ductility of casting alloys is usually low, and changes in the casting process as well as changes in the chemical composition and/or heat treatment which aimed at improving some properties, can sometimes decrease the material strength for structural applications. Therefore it is important to assess the effect of changes in the microstructure both on the ductility and the strength of the material [4]. Previous investigations have shown that, besides the grain size and the grain boundaries, other important metallurgic factors that significantly affect the ductile fracture of aluminum alloys are second phase particles inherently contained in the alloys, including large Fe, Cu, and Si-rich inclusions (about 1–10 μm in diameter), intermediate Cr, Mn, or Zr-rich dispersoids (about 0.05–0.5 μm in diameter), and small precipitates (nanometer size). The large particles, defined as constituents, are brittle in nature and are usually the primary void/crack initiators or the preferential crack propagation path. As a result, the influence of the

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constituents on the ductile fracture of aluminum alloys has aroused extensive attention [5].

The mechanical properties of heat-treated aluminum alloys are highly dependent on the thermal ageing as the strengthening precipitates are sensitive to both the ageing temperature θ and the ageing time t. Therefore in order to meet industrial requirements in terms of material properties, heat treatments have to be perfectly controlled to precisely manage the ageing process. As a consequence there has been an increasing interest in establishing precise quantitative relationships between the mechanical properties usually expressed in terms of Young modulus E and plastic flow limit σ_Y as a function of the processing parameters (θ, t) of the ageing treatment (see for instance [7]). An effective way to characterize these mechanical properties is to perform indentation experiments both as hardness and continuous indentation tests.

The aim of this work is to bridge the gap between ageing, microstructure and material properties for two aluminium alloys and to study, more particularly, the influences of thermal over-ageing on the mechanical behavior of LFC alloys by using indentation tests. In a first part, the materials and the experimental techniques are presented. The mechanical properties are then analyzed in terms of hardness at the macroscopic and the microscopic level. Properties of the different phases (intermetallics, eutectics) of the microstructure are then investigated. In the second part, the impact of the ageing on the mechanical properties is studied and modeled. In the last part, the microstructure and its evolutions are presented and the results of nanoindentation tests enable to determine the mechanical properties of eutectic and intermetallic phases.

2. Materials and investigation techniques

The alloys studied in this paper are two aluminium–silicon alloys commonly used in the automotive industry: an A319 without any heat treatment and an A356 with a T7 heat treatment. The chemical composition of both alloys is presented in Table 1. The T7 heat treatment consists in a thermal homogenization at 540 °C to dissolve the precipitates, followed by a water quenching and an artificial ageing at 200 °C during which the Mg_2Si precipitates are formed [9]. Both LFC alloys – A319 and A356 – were studied in non-aged and various ageing conditions to clarify the influences of over-ageing on the macroscopic mechanical behaviors.

The studied specimens were extracted from industrially produced parts in order to obtain a representative microstructure induced by the lost foam casting process. As shown in Fig. 1, they come from the firedeck intervalve zone of cylinder head prototypes. These specific zones are well known to be highly affected by thermomechanical loading and to be critical for fatigue cracks initiation. A preliminary characterization of the microstructure could be done in terms of Secondary Dendrite Arm Spacing (SDAS) and porosity. The SDAS is around 80 μm for LFC alloys, twice as large as the measured SDAS -close to 35 μm- in cylinder heads produced by a conventional DC process, which characterize a corser microstructure. Porosities and inclusions are more numerous and highly clustered while iron-, magnesium- and copper-containing intermetallics are found in the microstructure.

Table 1
Chemical composition of Lost Foam Cast A356 and A319.

Material	Si%	Mn%	Fe%	Mg%	Cu%	Zn%	Ti%	Ni%	V%	Zr%
A319	7.18	0.15	0.43	0.32	3.17	0.19	0.05	0.010	0.006	0.002
A356 (T7)	6.61	0.01	0.11	0.30	0.09	0.006	0.10	0.004	0.008	0.001

The specimens were submitted to different ageing histories both in terms of maximal temperature θ and ageing time t until reaching an eventual over-ageing state (steady stabilized). The over-ageing state has been determined by stabilization both in mechanical properties and microstructure composition of the material. For the studied A356 and A319 alloys, this state is observed in the condition of over-ageing equivalent to 500 h of heating at 250 °C (the industrial ageing time used at PSA Peugeot-Citroën). The tested ageing temperature levels were 150 °C, 200 °C and 250 °C and the ageing times were 3, 6, 9, 30, 60, 90, 200, 300 and 500 h. Each tested specimen corresponds to a specific ageing condition (θ, t). For optical and electronic microscopy observations, specimens have been polished with 500, 600, 1200 and 2400 grit papers (coarser polishing). Final polishing was carried out with 6, 3 and 1 μ m diamond pastes (fine polishing). Microstructural observations were conducted on areas of approximately $500 \times 500\ \mu m^2$ by using a Scanning Electron Microscope (SEM) S-3600N from Hitachi and a back-scattered electrons (BSE) technique.

Indentation experiments were performed simultaneously at different scales in order to test the global response of the aluminium alloys and to estimate both the aluminum matrix and the different phases mechanical properties separately at the microscopic level. The protocol sums up in:

(i) Vickers hardness experiments with an indentation depth δ of the order of 50 μm and an indentated area A around 100 μm^2, denoted next as *macro hardness*, are performed using a Test Well Vickers machine, with a 10 kg weight.
(ii) Berkovitch continuous indentation experiments with an indentation depth δ of the order of 1 μm and an indentated area A around 10 μm^2, denoted next as *nanoindentation*, are performed with a MTS XP nanoindentor machine using different measuring techniques. A simple hardness test, then a Depth Sensing Indentation (DSI) (see [10,11] for a general presentation) or a continuous stiffness measurement (CSM) (see [12–14]) are performed. The latter is characterized by a an oscillating displacement of 2 nm with a frequency of 45 Hz imposed to the indenter tip.

Vickers macro hardness tests were performed for different ageing times to estimate the evolution of macroscopic properties of the alloy with respect to the ageing state. 10 measurements were performed on the surface of each specimen and the mean value and standard deviation of macro hardness are presented in Table 2.

Nanoindentation tests were performed to evaluate the mechanical properties at the scale of the heterogeneities of the aluminum matrix and of the intermetallic/eutectic compounds completed with a careful microscopic observations before and after indentation. The nanoindentation technique has been used to measure the hardness H_B and to estimate the elastic modulus E of the intermetallic and eutectic phases. A variable maximum load was applied in order to achieve a high spatial resolution while still obtaining reliable indentation data. Tables 3 and 4 summarize the materials, ageing condition and number of indentations on each intermetallic and eutectic phase. The obtained results are discussed in the next sections, following two objectives. First, a macroscopic law is developed to describe the macro hardness evolution versus ageing time. A comprehensive study of the microscopic behavior of each of the component is then proposed with a peculiar attention on the ageing impact.

2.1. Effects of ageing on macro hardness

Fig. 2 represents the evolution of macro hardness as a function