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# Effect of natural aging on quench-induced inhomogeneity of microstructure and hardness in high strength 7055 aluminum alloy

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ABSTRACT

The effect of natural aging on quench-induced inhomogeneity of microstructure and hardness in high strength 7055 aluminum alloy was investigated by means of end quenching technique, transmission electron microscopy and differential scanning calorimetry thermal analysis. The hardness inhomogeneity in the end-quenched specimens after artificial aging decreases with the increase of natural aging time prior to artificial aging. The quench-induced differences in the amount and size of $\eta'$ phase are large in the end-quenched specimen after artificial aging at 120 °C for 24 h, leading to high hardness inhomogeneity. Natural aging for a long time results in a larger amount of stable GPI zones in the slowly-quenched sample, and thus decreases such differences in the end-quenched specimens after subsequent artificial aging, leading to lower hardness inhomogeneity. The hardness inhomogeneity can be reduced from 14% to be 4% by natural aging for 17,280 h prior to artificial aging.


## 1. Introduction

Al–Zn–Mg–Cu alloys are well-known precipitation hardening aluminum alloys and have been widely used as structural materials of aircrafts due to their high specific strength. In order to achieve high strength, the precipitation structure must be well controlled, and this depends on good understanding of the precipitation processes during aging, which is a critical step for production of these alloys. As a consequence, a number of investigations have been focused on the precipitation behavior in Al–Zn–Mg–(Cu) alloys in the past decades [1–9]. It is well known that during aging Guinier–Preston (GP) zones and/or metastable $\eta'$ phase precipitates, which have good hardening effect [1,2,8], can form from the super-saturated solid solution (SSSS). There are generally two kinds of GP zones, i.e., GPI zones and GPII zones, therefore the precipitation sequences shown below are most likely to occur during aging [3,4,10]:

1: SSSS → GPI zones → Metastable $\eta'$ phase → Stable $\eta$ ($MgZn_2$) phase
2: SSSS → VRCs → GPII zones → Metastable $\eta'$ phase → Stable $\eta$ ($MgZn_2$) phase

GPI zones are linked with solute-rich clusters and fully coherent zones with internal order described by AuCu(I)-type structure [11,12]; they can form over a wide temperature range. GPII zones are likely related to vacancy-rich clusters (VRCs) [3], and thus highly dependent on the vacancy concentration. GPII zones generally form after quenching from temperatures above 450 °C and aging at temperatures higher than 70 °C [12]; they can also appear after aging at lower temperatures for a very long time [5,6,13]. Some researchers thought that GPI zones can transform into metastable $\eta'$ phase [2,3,14], while others believed that GPII zones act as precursors to metastable $\eta'$ phase [12,15,16]. It is often accepted that the maximum hardening effect is associated with metastable $\eta'$ phase [2,4,8,17]. In general, the aging treatment of semi-products of Al–Zn–Mg–Cu alloys starts with natural aging after solution heat treatment and quenching. During natural aging, not only the size and number density but also the type of clusters and GP zones may change with time [6,18], and this can exert profound effect on the metastable $\eta'$ phase during subsequent artificial aging and consequently the final properties [1,18,19]. A better understanding of the microstructural evolution during natural aging is of importance in optimizing the precipitation structure and improving mechanical properties.

To date, most investigations were focused on ternary Al–Zn–Mg alloys and fewer on quaternary Al–Zn–Mg–Cu alloys. As Cu may have great effect on precipitation behavior [20–22], detailed investigations are still needed on the precipitation structure in

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Al–Zn–Mg–Cu alloys. Moreover, most previous investigations were carried out on alloys rapidly-quenched from solution heat treatment temperature into water, but fewer on alloys subjected to slow quenching. Slow quenching decreases the solutes and vacancy level in the solid solution [19,23], and thus lowers hardening potential and mechanical properties after aging [24–26]. In semi-products with large section, quenching rate in the mid-plane layer is often not high enough, and consequently the microstructure and properties are not uniform. For instance, in an aluminum alloy 7010 forging the yield strength was about 50 MPa higher at the corner than at the core location [27]. This phenomenon is easier to appear in alloys with high quench sensitivity. For practical applications, it is often desirable to decrease inhomogeneity in these semi-products, as the inner materials with lower properties are often exposed after subsequent machining for structural components. As shown previously the chemical compositions and microstructure can probably be optimized to decrease quench sensitivity so as to decrease inhomogeneity [24,28–30]. For a given alloy, it is possible to decrease microstructure and property inhomogeneity by increasing cooling intensity [31], but this may lead to high residual stress or even distortion [32,33]. Optimizing aging parameters is another probable way to decrease inhomogeneity. Appropriate aging can decrease quench sensitive effect significantly [34] and thus improve hardenability of Al–Zn–Mg–Cu alloy thick plate [35].

End quenching technique is a powerful tool to investigate the effects of chemical compositions, quenching rate and heat treating parameters for precipitation hardenable aluminum alloys [29,36]. By cooling one end of the specimen, a continuous decrease in the quenching rate along the longitudinal direction of the specimen can be obtained; this is similar to the quenching condition through the thickness of semi-products with large section. Therefore it is a good technique to investigate quench-induced microstructure and property inhomogeneity in Al–Zn–Mg–Cu alloy materials.

In this work, the effect of natural aging on quench-induced inhomogeneity of microstructure and hardness in high strength 7055 aluminum alloy thick plate was investigated using end quenching technique, and the mechanism was discussed based on microstructure examination by transmission electron microscopy (TEM) and differential scanning calorimetry (DSC) thermal analysis. 7055 aluminum alloy was selected for investigation because of its high quench sensitivity [37].

## 2. Experimental

The material was a 60 mm hot-rolled thick plate of 7055 aluminum alloy with chemical compositions (wt.%): Al–8.10Zn–2.08 Mg–2.25Cu–0.11Zr, Fe < 0.07, Si < 0.07. Specimens of 125 mm in length × 25 mm in width × 25 mm in thickness were cut from the plate for end quenching test. After solution heat treatment at 470 °C for 1 h in an air furnace, the specimen was taken out rapidly, placed in a fixture and cooled to room temperature by exposing at one end to a vertical stream of room temperature water. The average cooling rate through 420–230 °C was estimated to be about 1250 °C/min, 630 °C/min, 164 °C/min and 138 °C/min at the locations of 3 mm, 23 mm, 53 mm and 78 mm from the water-cooled end [35], respectively; beyond 78 mm the cooling rate tended to be a constant value of about 135 °C/min. Then, some specimens were naturally aged at room temperature up to 17,280 h, other specimens were artificially aged at 120 °C in an oil bath for 24 h after natural aging for 0 h, 4320 h, 8640 h and 17,280 h, respectively.

The aged specimens were evenly cut into two parts and the hardness on the center layer was measured after grinding and polishing. The Vickers hardness testing was performed on an HV-10B hardmeter with a load of 3 kg and five measurements were made to obtain an average value. Because of the time for preparing hardness samples, the hardness after natural aging for about 2 h was regarded as the as-quenched hardness. Thin samples at different distances ($d$) from the water-cooled end in the end-quenched and aged specimens were cut for microstructure study. DSC thermal analysis was conducted on a NETZSCH DSC 200 F3 to characterize precipitates in the samples. The sample size was about 5 mm in diameter and 0.3 mm in height, and the weight was about 17 mg. High purity aluminum (99.99%) was used as reference. A baseline scan was recorded using a high purity aluminum (99.99%) sample and subsequently subtracted from the alloy sample scans. The samples were heated from room temperature to 500 °C with a heating rate of 10 °C/min under an argon atmosphere. Three samples were tested to confirm the results were repeatable. Foils of 3 mm in diameter, 0.08 mm in thickness were prepared and electropolished in 20% $HNO_3$ + 80% $CH_3OH$ solution below −20 °C, and then observed on a FEI TECNAI G$^2$ 20 TEM operated at 200 kV to examine precipitation structure in the aged samples.

## 3. Results

### 3.1. Hardness inhomogeneity

The hardness in the end-quenched specimen after natural aging is plotted as a function of the distance from the water-cooled end in Fig. 1(a). The hardness of the as-quenched specimen (natural aging for about 2 h) is quite low, about 110 HV3, and does not change much with the increase of distance, i.e., there is little effect of cooling rate on the hardness. Due to the time required for specimen preparation, it was difficult to measure the hardness immediately after end quenching, so it is unlikely to tell the increment of hardness during natural aging for 2 h. The specimens were obviously hardened after natural aging for a quite long time, and the hardness over the whole distance increased with time. After about 12960 h, natural aging seems to have little influence on hardness, and the value tends to be constant, about 192 HV3. With distance increasing from the water-cooled end, the hardness tends to decrease after natural aging. This can be attributed to the decrease of cooling rate and hence lower hardening effect with the increase of distance. In order to describe the effect of natural aging on the hardness inhomogeneity more clearly, the hardness retention value (HRV) at different distances was calculated by:

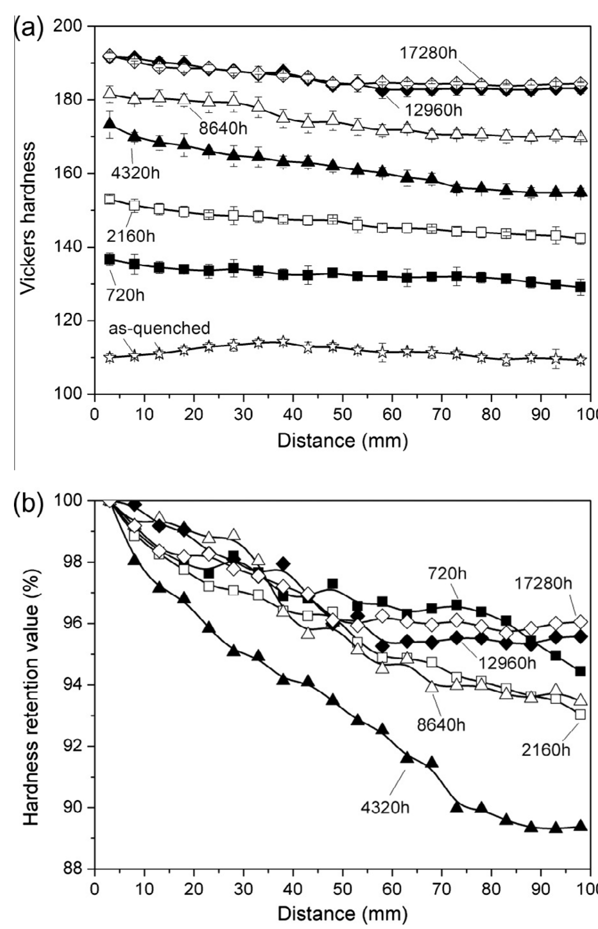


**Fig. 1.** Influence of natural aging time on the (a) hardness and (b) hardness retention value in the end-quenched specimens after natural aging.