Chapter 14
Anelasticity in Al-Alloy Thin Films: A Micro-mechanical Analysis

J.P.M. Hoefnagels, L.I.J.C. Bergers, and M.G.D. Geers

Abstract Micro-electromechanical systems enable many novel high-tech applications. Aluminum alloy thin films would be electrically favorable, but mechanical reliability forms fundamental challenges. Notably, miniaturization reveals detrimental time-dependent anelasticity in free-standing Al-alloy thin films. Yet, systematic experimental studies are lacking, perhaps due to challenges in microscale testing.

To this end, a microbeam bending methodology (with $<4\ \mu\varepsilon$ strain resolution) and nano-tensile tester (with $\sim$70 nN and $<6\ \mu\varepsilon$ resolution) have been developed for reproducible long-duration characterization of anelasticity of on-wafer 5 μm-thick Al-(1 wt%)Cu test structures under real-time in situ microscopy. Time-dependent anelasticity was indeed observed, both in bending and in tension, and a multi-mode visco-elastic model was found to described and predict the non-linear anelastic behavior between $\sim$1 and $\sim$105 s.

To gain insight in the underlying micro-mechanisms, time-dependent anelasticity was characterized under systematic variation of the grain boundary density and copper precipitate state/distribution (carefully analyzed using HRTEM/EBSD/WAXD/EDX/SEM), yielding a wealth of information. Surprisingly, both microstructural features were excluded as the (primary) cause of the time-dependent anelasticity. Based on dynamic strain aging effects observed in nano-indentation, an underlying micromechanism responsible for time-dependent anelasticity was hypothesized.

Keywords MEMS • Anelasticity • Micro-mechanics • Microscopic analysis • In situ mechanical testing

14.1 Introduction

Micro-electromechanical systems (MEMS) enable novel high-tech applications in, e.g., aerospace, biomedicine and wireless communications by integrating electrical and mechanical functionalities to address increasing demands: higher performance, more functionality, smaller dimensions. Although aluminum alloy thin films are electrically favorable, the compromised mechanical reliability still forms a fundamental challenge. Miniaturization has revealed detrimental size-dependent behavior in time-dependent elasticity, i.e., anelasticity, for free standing Al-alloy thin films, see Fig. 14.1. However, systematic experimental analyses of these mechanics have yet to be performed, partly due to the challenges of microscale testing. This work therefore aimed to develop on-wafer mechanical characterization methods and to acquire insights into the underlying physical micro-mechanisms responsible for anelastic size-effects.

14.2 On-Wafer Mechanical Tests for Characterization of Long-Term Anelasticity

Two methodologies were developed for reproducible characterization of anelasticity of on-wafer test structures in combination with microscopy. The specimens were 5 μm-thick aluminum and Al-(1 wt%)Cu films fabricated in a MEMS fabrication process. A microbeam bending methodology [1, 2], see Fig. 14.2, and a nano-tensile test methodology [3],

J.P.M. Hoefnagels (✉) • L.I.J.C. Bergers • M.G.D. Geers
Department of Mechanical Engineering, Eindhoven University of Technology, Den Dolech 2, Eindhoven, 5612AZ The Netherlands
e-mail: j.p.m.hoefnagels@tue.nl

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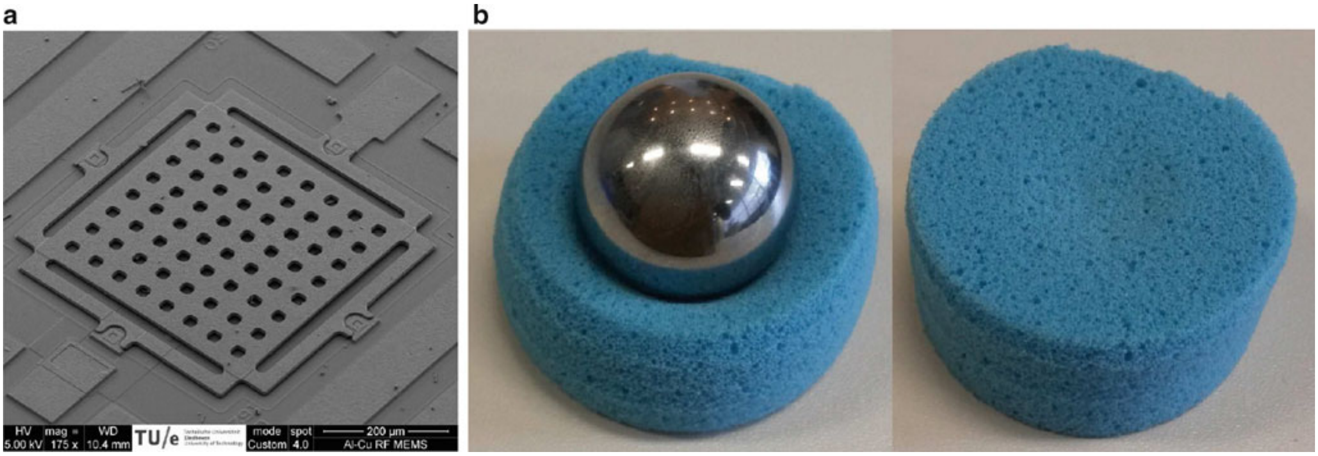

Fig. 14.1 (**a**) A scanning electron microscope image of a radio-frequency microelectromechanical system: a capacitive switch consisting of a perforated square plate suspended at its corners by hinging beams that are fixed to four points on the chip substrate. The plate and beams are made of an aluminum-alloy. It suffers from time-dependent anelasticity, which is commonly known in polymers as visco-elasticity as demonstrated by polymer foams, known as memory foam, (**b**) that recover their shape slowly after being deformed

see Fig. 14.3, were developed. To measure minute strains involved in anelasticity, both methodologies relied on novel improvements and developments to optical imaging and processing techniques: digital holographic microscopy, confocal optical profilometry, bright field optical microscopy, global digital image correlation. This ensured reproducible deformation measurements of $<6 \times 10^{-6}$ strain during time spans of days. The microbeam bending method relied on a multi-mode anelasticity model to yield key characteristics of the mechanical behavior without an explicit force measurement. For the nano-tensile testing drift-compensated force measurements achieved resolution down to 70 nN and maximum forces up to 200 mN. Hence, the two micromechanical techniques enabled characterization of anelasticity of these thin metal films.

14.3 Characterization of Al-(1 wt%)Cu Specimens [4]

Subsequently, the microstructure of pure aluminum and Al-(1 wt%)Cu thin films were characterized to investigate the influences of grain boundaries and alloy microstructure, i.e., solute concentration, precipitate type, size and distribution [4]. The grain boundaries of various microstructures were characterized with electron backscatter diffraction. The influence of precipitates was investigated by altering their state through heat treatments for homogenization and aging. Backscatter electron contrast imaging, energy dispersive spectroscopy, wide angle X-ray diffraction and high resolution transmission electron microscopy [4] revealed the variations of precipitate type and distribution in the Al–Cu alloy microstructure. Interestingly, the precipitation in the thin films was different compared to precipitation in bulk. The precipitation appeared to occur sooner and did not yield θ' precipitates. This difference was hypothesized to be due to preferential nucleation of θ at the grain boundary grooves and surface, see Fig. 14.4, which could result in a lower free energy of the system with respect to bulk precipitation.

14.4 Results on Time-Dependent Anelastic Mechanics [5]

Finally, the two mechanical characterization techniques were applied to measure the time-dependent anelastic response as function of the microstructural variations [5]. The influence of grain boundaries and precipitation state was probed with the microbeam bending methodology on the Al–Cu specimens. However, results suggest a negligible influence of either grain boundaries or precipitate size or state, see Fig. 14.5. Nano-tensile tests on Al–Cu and pure Al revealed nearly identical anelastic behavior as compared to the Al–Cu microbeams. These observations suggest that the grain boundaries, precipitates and even Cu-solutes did not influence the time-dependent anelasticity [5]. This led to the hypothesis that the underlying micromechanism responsible for time-dependent anelasticity could be related to dislocation junctions and entanglements and their diffusion-limited formation and relaxation.

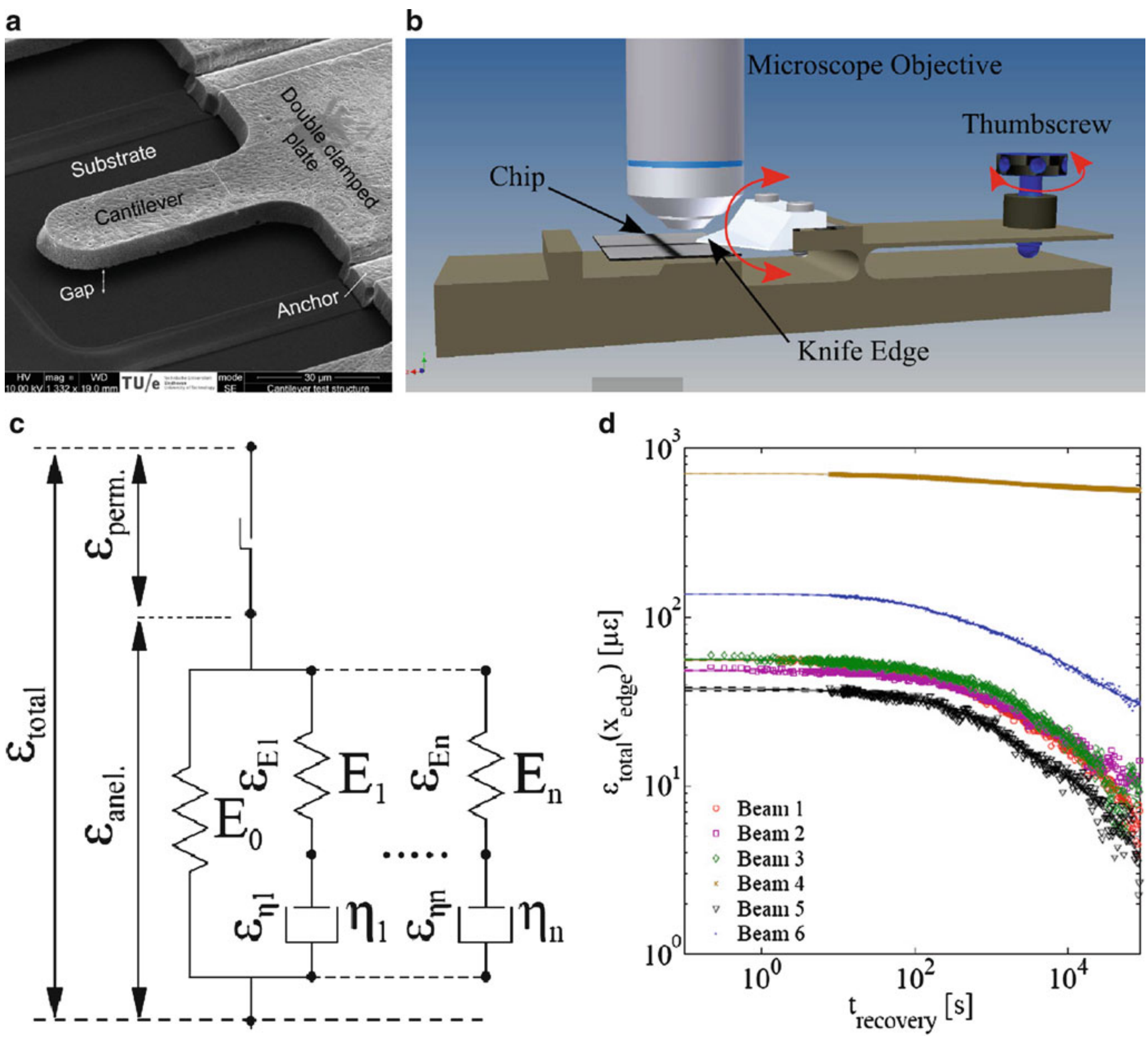

Fig. 14.2 The microbeam bending methodology [1, 2]: (**a**) A SEM-image of a test cantilever beam that is attached to a free-standing plate clamped on three sides by an anchor. (**b**) Schematic side view of the micro-clamp used to deflect on-chip cantilevers with high resolution under a profilometer. Thermal and mechanical drift effects in the load are minimized through a dedicated thermomechanical design. (**c**) Time-dependent behavior described by a standard solid multi-mode viscoelastic model with plastic dissipation resulting in permanent deformation. (**d**) Strain recovery evolution with individual model responses

14.5 Conclusions

A microbeam bending methodology (with <4 $\mu\varepsilon$ strain resolution) and nano-tensile tester (with ~70 nN and <6 $\mu\varepsilon$ resolution) have been developed for reproducible long-duration characterization of anelasticity of on-wafer 5 μm-thick Al-(1 wt%)Cu test structures under real-time in situ microscopy. Time-dependent anelasticity was indeed observed, both in bending and in tension, and a multi-mode visco-elastic model was found to described and predict the non-linear anelastic behavior between ~1 and 10^5 s. Time-dependent anelasticity was carefully analyzed characterized under systematic variation of the grain boundary density and copper precipitate state/distribution. Surprisingly, both microstructural features were excluded as the (primary) cause of the time-dependent anelasticity. An underlying micromechanism responsible for time-dependent anelasticity was hypothesized.

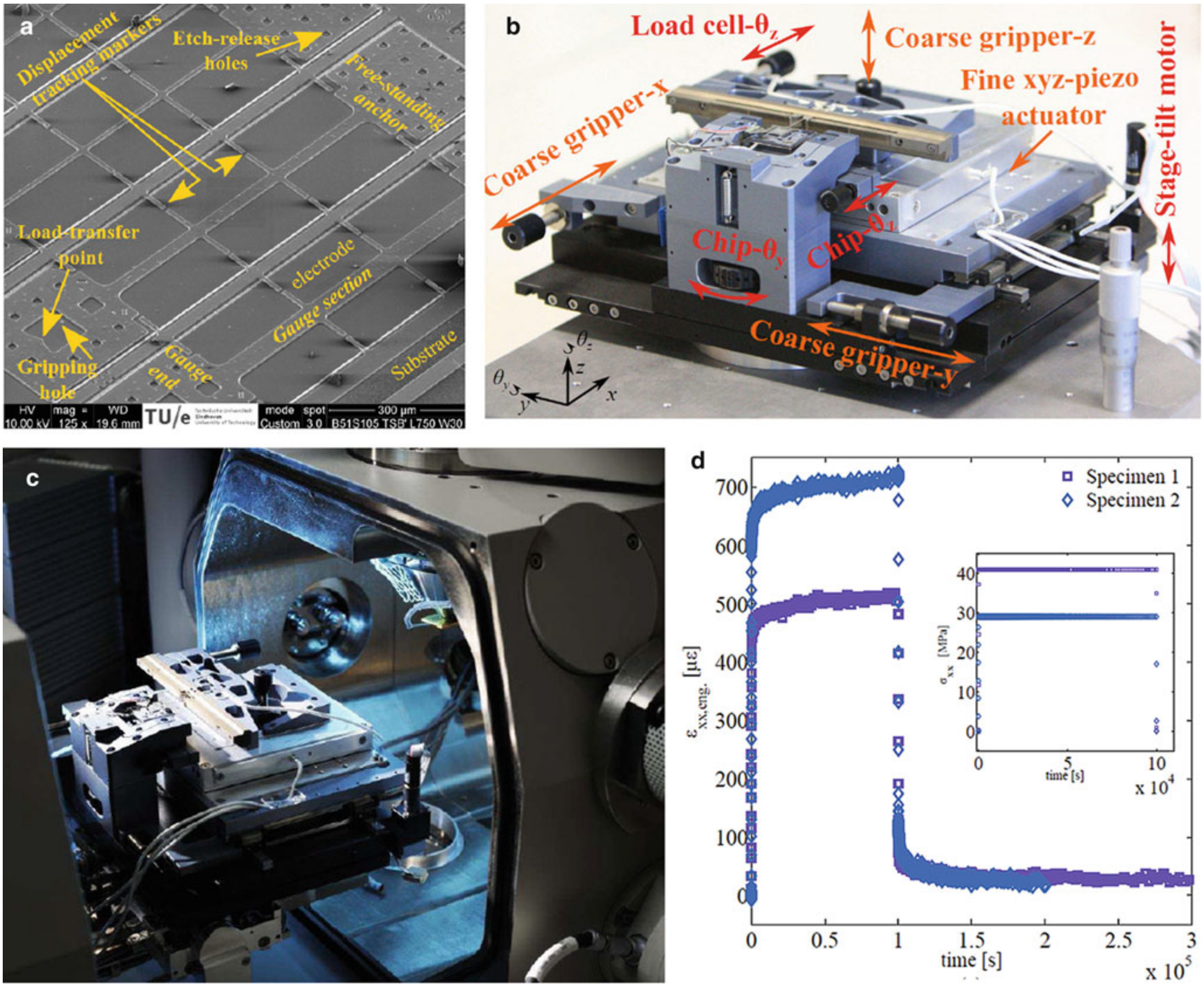

Fig. 14.3 The nano-tensile test methodology [3]: (**a**) Scanning electron micrograph of a free-standing tensile specimen that is fixed at one end along the perimeter of the anchor. At the free-standing gauge-end a gripping hole is made with load-centering feature. Along the substrate and the length of the beam several displacement tracking markers are fabricated to measure the substrate displacement and the gauge deformation. (**b**) Overview of the nano-tensile tester highlighting manual precision manipulators for angular alignment, coarse xyz-positioning and a xyz-piezo actuator for nm-positioning. (**c**) Nano-tensile stage placed in a FEI Quanta 600 ESEM. (Photo courtesy of Bart van Overbeeke Fotografie.) (**d**) Tensile creep experiment at constant stress (see inset) followed by time dependent anelasticity recorded for a tensile specimen

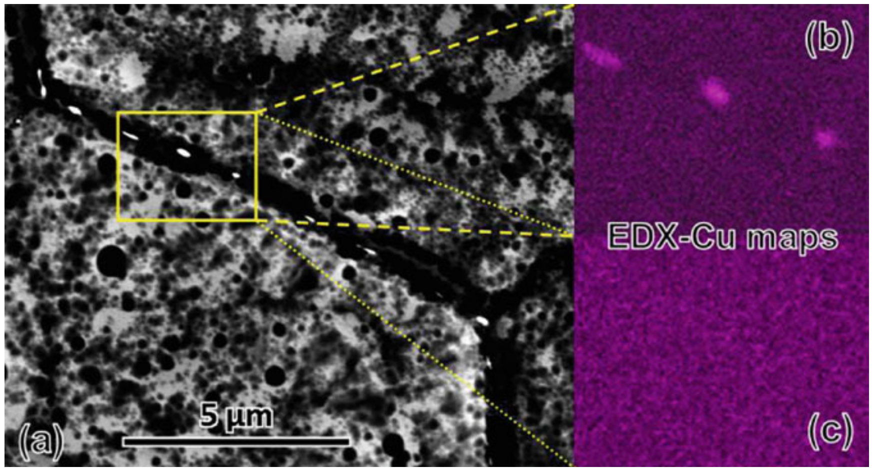

Fig. 14.4 The effect of the homogenization step at 550 °C on the precipitate state in the as-received Al-(1 wt%)Cu material. (**a**) back-scatter electron (BSE) image of large θ precipitates (with sizes up to half a micrometer) aligned on a grain boundary. (**b**, **c**) EDS maps of the copper concentration (**b**) before and (**c**) after the solution heat treatment, showing that all copper has been dissolved. BSE and EDS performed in a FEI Sirion FEG-SEM with EDAX EDS system and Apollo SDD detector. BSE obtained with Ebeam = 15 kV, EDS measured with Ebeam = 20 kV

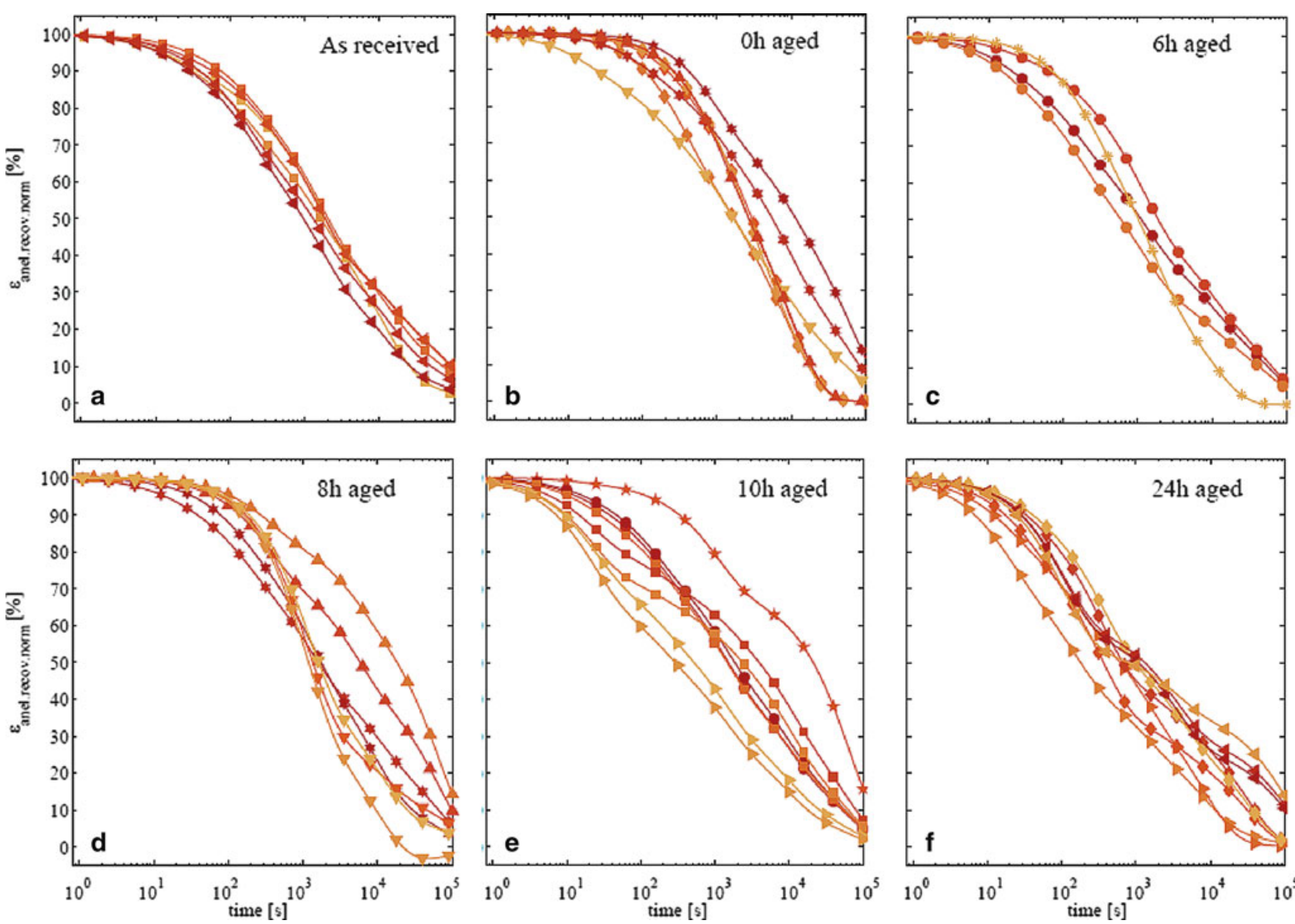

Fig. 14.5 Influence of the grain boundary density on the normalized anelastic recovery evolution for specimens (**a**) as-received and (**b**) 0 h, (**c**) 6 h, (**d**) 8 h, (**e**) 10 h and (**f**) 24 h aged condition. The color intensity (ranging from *light orange* to *dark red*) corresponds to the increase in grain boundary density, ρ'_{GB}. These results suggest a negligible influence of either grain boundaries or precipitate size or state (Color figure online)

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