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ORIGINAL ARTICLE

Evaluation of roughness, hardness, and strength of AA 6061 molds for manufacturing polymeric microdevices

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Abstract In the manufacturing of polymeric microfluidic devices, micro-molds play a key role because they determine not only the manufacturing cost but also the quality of the molded parts. Recently, a high-quality aluminum alloy 6061 (AA6061) mold with fine features less than its grain size has been fabricated economically by a hot embossing technique. However, temperature cycling during hot embossing process in mold manufacturing reduces significantly the original tensile strength and hardness of the AA6061-T6 alloy substrate, which is not desirable. In this study, a tempering process is carried out to recover the tensile strength and hardness of the embossed mold. To evaluate the changes of these properties, surface roughness, tensile strength, and hardness values were measured in each stage: (1) before hot embossing, (2) after hot embossing, and (3) tempering to T4 and tempering to T6. The results obtained demonstrate that the original strengths and hardness can be fully recovered by a post-tempering process after hot embossing, but with an increase in surface roughness. Moreover, accelerated testing was carried out to evaluate the changes in hardness and roughness of AA6061-T4 and T6 molds under the typical hot embossing temperature cycles of manufacturing polymeric devices. The results obtained indicate that these temperature cycles have only a minor effect on the roughness of both T4 and T6 molds and will increase the hardness of T4 molds to T6 temper, and have negligible effect on the hardness of a T6 temper mold.

Keywords Micro-mold · Hot embossing · Heat treatment · Aluminum alloy 6061

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1 Introduction

Microfluidics is the manipulation of fluids in channels having at least two dimensions at the micron scale [1]. Microfluidic devices were originally manufactured with glass and silicon by conventional, planar fabrication techniques—photolithography and etching—in the early 1990s [2]. Recently, due to their ease of fabrication and low cost, polymers are employed as base materials in making microfluidic devices [3]. Polymeric microfluidic devices can be found in capillary electrophoresis, multicomponent reactions, bio-sensors, genetic analysis, or employed to measure parameters such as molecular diffusion coefficients, fluid viscosity and density, pH, and those related to reaction kinetics. Among the various methods to fabricate these devices are laser photo-ablation, X-ray photolithography, soft lithograph, hot embossing, and injection molding [3], with hot embossing and injection molding being the two most commonly used methods. Although these two manufacturing processes are different, they follow similar principles: the final products are replications of the master or mold, which has an important role in determining both the quality and the manufacturing cost of the final devices.

Silicon molds are generally fabricated using wet-chemical etching procedures or by deep reactive ion etching (DRIE), which can provide structures with relatively high

aspect ratios and arbitrary shape with vertical sidewalls [4]. However, silicon molds are not feasible for mass production purpose as they can break easily due to their inherent brittleness when there is slight misalignment of mold and substrate. As an alternative, strong and tough metallic molds with longer lifespan can be fabricated by high precision micromachining (micromilling [5, 6], micro electrical discharge machining (μEDM) [7, 8], laser micromachining [9–11], and micro electro chemical micromachining (μECM) [12]); lithography, galvanoforming, and plastic molding (LIGA) [13]; or by cold and superplastic embossing [14]. The surface finish of molds manufactured by micromilling and μEDM methods is relatively higher than that by the lithography method (0.3 μm for micromilling and 0.4–0.5 μm for μEDM [15]). In addition, micromilling method has some disadvantages in that it could not produce sharp inside corner and there is limitation on the minimum feature size fabricated [5]; the drawbacks of μEDM are the slow material-removing rate and the rough sidewall of microstructures [16]. μECM can create high surface finish features [17], but its major disadvantages are the very high machine and tooling cost [18] as well as the very low metal removal rate [19]. LIGA method can provide high-aspect-ratio microstructures with smooth sidewalls and submicron features, but the process is costly and sophisticated [13]. Cold embossing and superplastic embossing are easy-to-apply methods, but they cannot create microchannels with sharp edges [14]. Polymer molds made by soft lithography [20] or two-stage hot embossing [21, 22] can also be employed in the manufacturing of microfluidic devices; however, the intrinsic limitations of polymeric molds, such as their low melting temperature and strength as compared to the metallic molds, are the main drawbacks of these techniques. Lately, bulk metallic glass (BMG) molds were fabricated by hot embossing method from a silicon master [23]. With very high strength, endurance, and good surface finish, BMG molds are ideally suited for the mass production of polymeric microfluidic devices, except that BMG is an expensive material which limits its wide adoption. To have a mold with sufficient strength with acceptable cost, we recently fabricated AA6061 molds by hot embossing using a silicon master [24, 25]. The feasibility of employing AA6061 mold to fabricate polymeric microdevices has been demonstrated through the successful fabrication of microchannels on TOPAS 8007 substrate [24]. However, major concerns remain on the strength and roughness of the mold.

Surface roughness, hardness, and strength are the main criteria for the evaluation of a metallic mold. Due to the temperature cycles of the hot embossing process, these properties of AA6061 mold change significantly as compared to the as-received AA6061-T6 substrate. To recover the mold's strength and hardness to the original as-received AA6061-T6 condition, a tempering process can be carried out. All the values of strength, hardness, and roughness before embossing (i.e., as-received), after embossing, and tempering to T4 and T6 are examined in this study. In the subsequent manufacturing of microdevices using the AA6061 molds, the AA6061-T4 and AA6061-T6 molds will experience additional temperature cycles which may affect their hardness and the roughness values. Thus, accelerated testing was employed to evaluate these possible changes.

2 Experimental procedure

2.1 Equipments

Hot embossing on AA6061 specimens with silicon masters and tensile testing were carried out using an Instron 8502 testing machine equipped with an air furnace which can reach up to 1,000°C. Two thermocouples were attached to the hot compression platens for controlling the hot embossing temperature. PLμ Confocal Imaging Profiler was employed to measure the surface roughness of AA6061 specimens. Hardness of AA6061 specimens before and after hot embossing and after tempering was measured by a FutureTech FM-300E Vickers Hardness Tester. Tempering process was carried out with an Elite Furnace for soaking at 529°C and a Venticell Oven for artificial aging.

2.2 AA6061-T6 specimens

AA6061-T6 is a precipitation hardened aluminum alloy, containing 0.84% magnesium and 0.7% silicon as its major alloying elements. Two types of specimens were prepared respectively for hot embossing and for tempering.

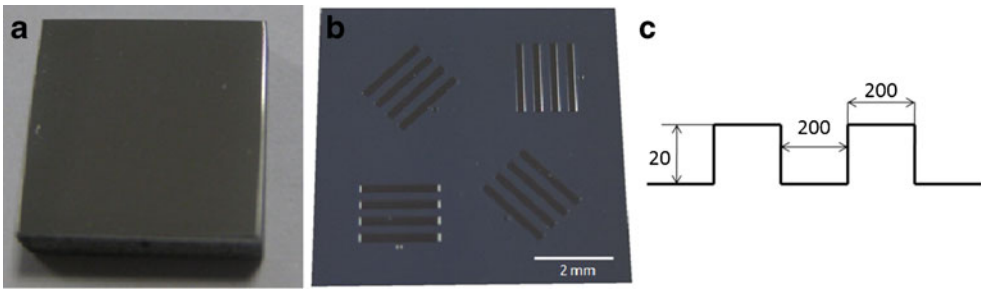

Fig. 1 **a** 8×8×3 mm polished AA6061-T6 specimen. **b** 8×8 mm silicon master. **c** Dimensions of microchannels on silicon master

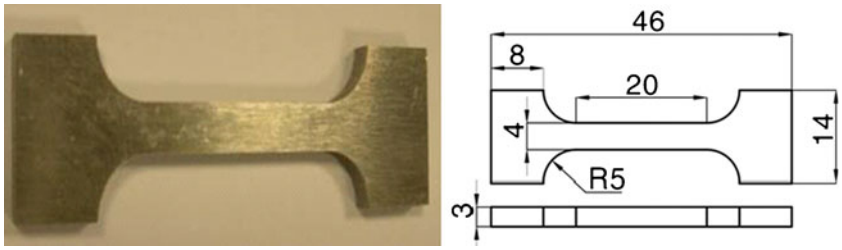

Fig. 2 Dog-bone shaped specimen for tensile test (unit, millimeters)

The 8×8×3 mm AA6061-T6 specimens for hot embossing were wire-cut from 3-mm-thick rolled AA6061-T6 sheet. These pieces were polished using SiC papers: grit 800, 1,200, 2,400, and 4,000 and then followed by 6- and 1-μm diamond pastes on polishing cloths. Figure 1a shows a typical hot embossing specimen.

The dog-bone specimens for tensile test were wire-cut from rolled AA6061-T6 sheet, with the dimensions of a typical specimen shown in Fig. 2

2.3 Silicon master

Silicon masters were fabricated from a 500-μm thick silicon wafer. The manufacturing process includes three major steps: photolithography, DRIE, and dicing of wafer to 8×8 mm square pieces. A typical silicon master is shown in Fig. 1b. The microchannels on a silicon master have a width of 200 μm, a depth of 20 μm, and a distance of 200 μm between the edges of the channels as shown in Fig. 1c.

2.4 Hot embossing process

A sandwich of AA6061 specimen and silicon master was put in between the top and the bottom platens where they were heated to 500°C. A compressive load of 1,400 N was then applied with an embossing rate of 0.1 mm/min. The silicon master was subsequently demolded manually after air cooling. Details of the hot embossing process can be found in [24].

2.5 Tempering process

The temperature cycle of the hot embossing process causes a loss of properties of AA6061-T6 alloy, resulting in a reduction of strength and hardness. To recover the lost strength and hardness, a T6 tempering process can be carried out on the hot embossed AA6061-T6 specimens.

Tempering is a heat treatment technique that can be applied to precipitation hardenable aluminum alloys. The AA6061 alloy was held at their solutionizing temperature for a considerable amount of time to ensure a complete solutionizing and homogeneous phase. It is then quenched rapidly such that the intermetallic compounds can remain in the saturated solution. The natural and artificial aging steps are employed subsequently to precipitate out the intermetallic compounds in smaller particles which disperse homogeneously. This results in a stronger and harder alloy through impeding the dislocation movement along the slip planes.

The T6 tempering process [26] includes four steps:

- Soaking specimen in a furnace for 40 min at a temperature of 529°C
- Water quenching specimen immediately after it is taken out from the furnace
- Natural aging by leaving the specimen at room temperature for 96 h to get T4 temper
- Artificial aging by heating the specimen in an oven at 177°C for 10 h to achieve T6 temper

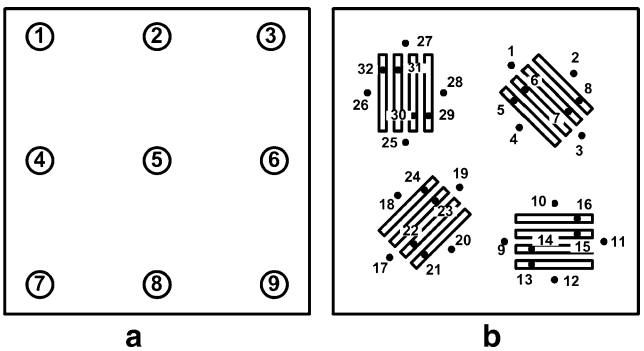

Fig. 3 Surface roughness measurement locations on **a** AA6061-T6 specimen before hot embossing and **b** AA6061 specimen after hot embossing and after tempering. Channel length and width are 2 mm and 200 μm, respectively

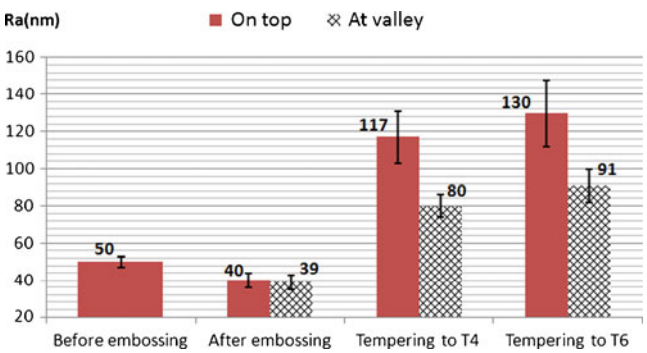

Fig. 4 Average roughness and standard deviation of all locations on top surface and at bottom of valleys of microchannels of three specimens before and after hot embossing, after T4 and T6 tempering

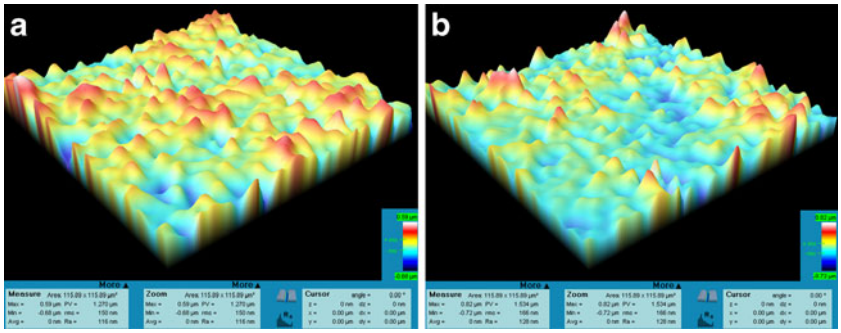

Fig. 5 Surface roughness on the top surface of AA6061 mold **a** after tempering to T4 and **b** after tempering to T6, measured by PLμ Confocal Imaging Profiler

3 Results and discussions

3.1 Surface roughness measurements

Before embossing, roughness values were measured at nine different locations as shown in Fig. 3a. After hot embossing and after tempering, surface roughness at 16 locations on the top surface of the specimen and 16 locations at the valleys of the embossed channels were measured, see Fig. 3b.

The average surface roughness of the three specimens at the nine different locations before hot embossing had only minor and insignificant variations between locations and specimens. The average value for all the nine locations was 50 nm with a standard deviation of 3 nm. After hot embossing, the average roughness on the top surface for all the 16 locations of the three specimens was 40 nm with a standard deviation of 4 nm, with minor and insignificant variations between locations and specimens. Similarly, the average roughness at the valleys of the channels was 39 nm with a standard deviation of 4 nm. In the hot embossing process, the silicon master was pressed onto the surface of AA6061 with a high load at high temperature. This would level out the roughness peaks on the top surface of the AA6061 specimen. Thus, hot embossing is likely to decrease the surface roughness of the specimen. After natural aging to T4 temper, the average roughness on the top surface and at the bottom of the valley increased rapidly to 117 nm (standard deviation of 14 nm) and 80 nm (standard deviation of 6 nm), respectively. After T6 tempering, the average roughness on the top surface increased further to 130 nm with a standard deviation of 18 nm; in contrast, the average roughness at the bottom of the valley only increased slightly to 91 nm with a standard deviation of 9 nm. These results are summarized in Fig. 4. It should be highlighted that despite the increase of surface roughness after tempering (to T4 or to T6), the roughness of the tempered specimens was less than the expected roughness of mold manufactured by micromilling (0.3 μm) or μEDM (0.4–0.5 μm). Figure 5 shows typical examples of surface roughness on the top surface of AA6061 mold after tempering to T4 and T6.

3.2 Hardness measurements

Before hot embossing, hardness values of three specimens were measured at nine selected locations as shown in Fig. 6a. After hot embossing and after subsequent tempering, the hardness was measured at nine selected locations as shown in Fig. 6b.

In each stage, before hot embossing, after hot embossing, after T4 tempering, and after T6 tempering, the average hardness of the three specimens at the nine different locations had only minor and insignificant variations between locations and specimens. Figure 7 shows the average and standard

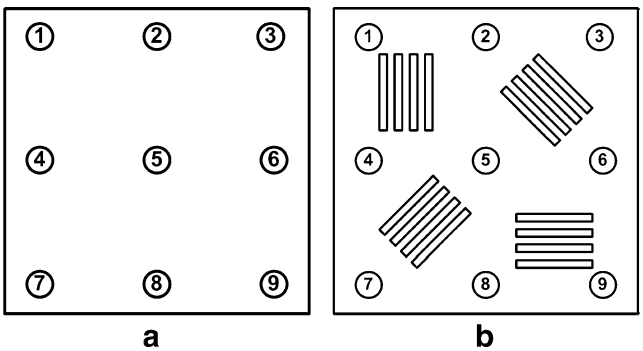

Fig. 6 Hardness measurement locations on **a** AA6061-T6 specimen before hot embossing and **b** AA6061-T6 specimen after hot embossing and after tempering. Channel length and width are 2 mm and 200 μm, respectively

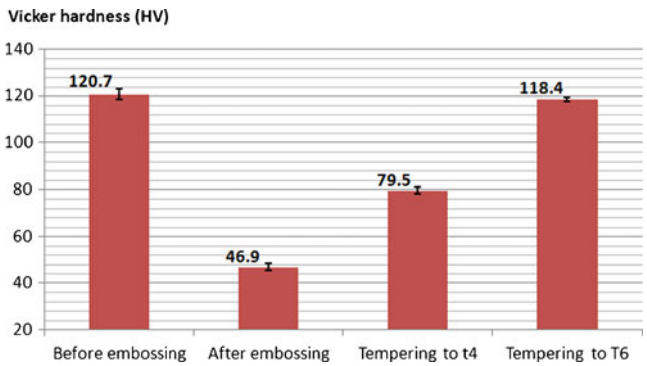

Fig. 7 Average and standard deviation of hardness of three specimens at nine locations before hot embossing, after hot embossing, after T4 and T6 tempering

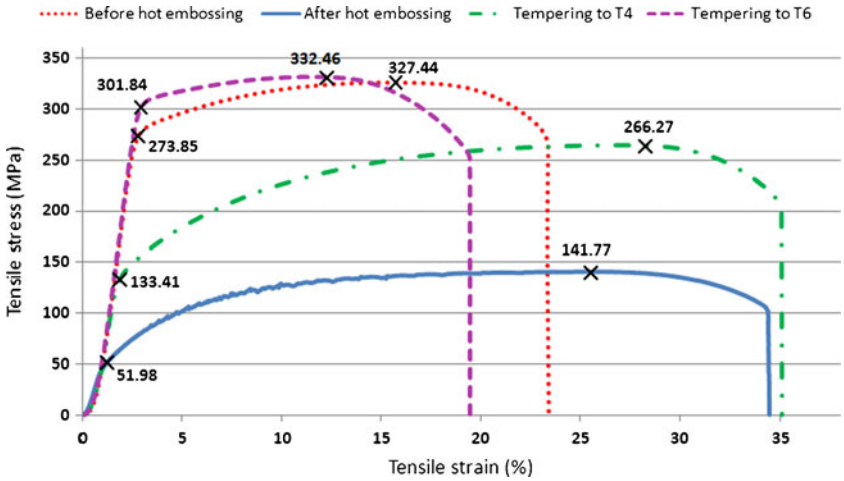

Fig. 8 Tensile tests of AA6061 in stages: before hot embossing, after hot embossing, tempering to T4, and tempering to T6

deviation of hardness values of AA6061 at all stages. Before hot embossing, the average hardness for all nine locations of the three specimens was 120.7 Vickers Pyramid Number (HV) with a standard deviation of 2.2 HV. After hot embossing, the average hardness for all nine locations of the three specimens reduced sharply to 46.9 HV with a standard deviation of 1.6 HV. The average hardness was partially recovered to 79.5 HV with a standard deviation of 1.5 HV after T4 tempering. After T6 tempering, the average hardness was fully recovered to 118.4 HV with a standard deviation of 0.8 HV.

When AA6061 is in T6 temper, the intermetallic precipitations are small particles dispersed on many slip planes [27]. These particles are obstructions which will impede effectively the dislocation movements resulting in improved strength and hardness of AA6061. As such, AA6061 before hot embossing (which is in the T6 tempering state) and after T6 tempering have high average hardness. In contrast, during the hot embossing process, AA6061 specimen was heated to 500°C, which is near its solutionizing temperature of 529°C. At this temperature, most of the intermetallic compounds would be dissolved in the solid solution. During the slow cooling phase of the embossing process, these compounds would precipitate out as relatively large particles and would not impede effectively the dislocation movements along the slip planes. This resulted in a considerable decrease of average hardness for all locations of all the three specimens after hot embossing. Compared to natural aging, artificial aging process can precipitate out the intermetallic compounds homogeneously. As such, the resulting average hardness after T6 tempering would be higher than that after T4 tempering.

3.3 Tensile strength measurements

Three specimens were tested in each stage: before hot embossing, after hot embossing, natural aging to T4, and artificial aging to T6. It should be noted that the so-called hot embossed specimens for tensile testing did not undergo the hot embossing process. Instead, they only experienced the temperature cycle of the normal hot embossing process but without any features embossed onto the specimen. This is to facilitate the subsequent tensile tests, as embossed fine features are not desirable features on tensile specimens.

Figure 8 shows the tensile tests of AA6061 before and after hot embossing, after T4 tempering, and after T6 tempering. Each curve was an average of three tensile test curves of three specimens. The × markers on these graphs show the locations of yield stress and ultimate tensile strength (UTS), and the numbers next to them show the average of these values of three specimens in different stages.

Before hot embossing, the AA6061 specimens were in T6 temper. The average yield stress and UTS were 273.85 MPa (standard deviation 0.23 MPa) and 327.44 MPa (standard deviation 0.12 MPa) respectively. After hot embossing, these values dropped dramatically to 51.98 MPa (standard deviation 0.35 MPa) for yield stress and 141.77 MPa (standard deviation 0.16 MPa) for UTS. Natural aging to T4 temper recovered partially the yield stress to 133.41 MPa (standard deviation 1.02 MPa) but recovered more substantially the UTS to 266.27 MPa (standard deviation 0.66 MPa). After

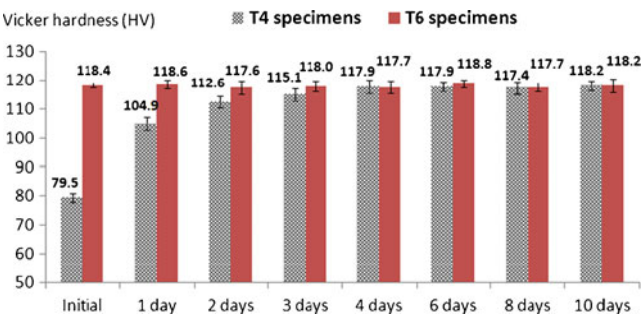

Fig. 9 Evolution of hardness of AA6061-T4 and T6 specimens during accelerated testing

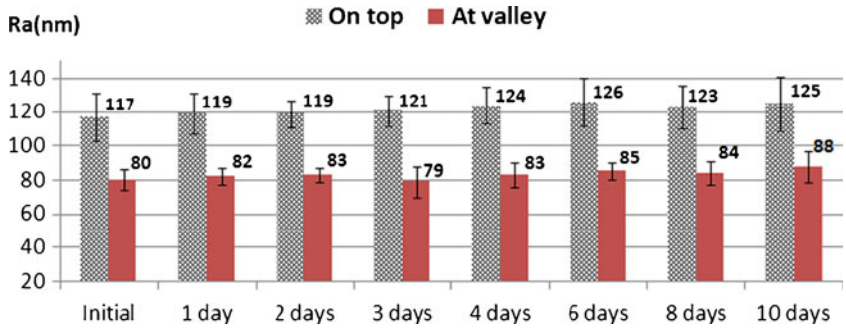

Fig. 10 Evolution of roughness of AA6061-T4 specimens during accelerated testing

artificial aging to T6 temper, the yield stress and UTS were fully recovered to 301.84 MPa (standard deviation 0.67 MPa) and 332.46 MPa (standard deviation 0.96 MPa) respectively. The reason for the considerable decrease after hot embossing and subsequent recovery after tempering of yield stress and UTS is similar to that for the loss and recovery of hardness as explained in Section 3.2.

3.4 Accelerated testing

The AA6061 molds will be subsequently employed to manufacture polymeric microdevices. One of the most widely used manufacturing methods is hot embossing. In this method, the AA6061 molds will have to experience temperature cycles of the manufacturing process which will affect the hardness and roughness of the molds. In order to document how roughness and hardness of these molds may change under temperature cycles, accelerated tests were carried out. Accelerated testing is a process of using the molds in an environment which is more severe than that of normal use so that all the changes in mechanical properties of the molds can be assessed quickly.

The usual hot embossing temperature for a polymer substrate will be near its glass transition temperature (T_g). The T_g for commonly used polymers is 150°C or less, e.g., polymethylmethacrylate 85–110°C, polycarbonate 140–150°C, polystyrene 74–109°C, polyvinyl chloride 75–105°C, and polyethylene terephthalate 73–80°C [28]. As such, the soaking temperature for the accelerating tests was chosen as 150°C. In these accelerated tests, AA6061 molds in both T4 and T6 temper were soaked in the furnace for 10 days at a constant temperature of 150°C. After every 24 h, three specimens in each temper were taken out to measure hardness and roughness. The roughness and hardness value are measured at selected locations as shown in Figs. 3 and 6, respectively.

The evolution of hardness of T4 and T6 specimens during accelerated testing is shown in Fig. 9. The hardness of T4 specimens increased steadily from 79.5 HV (standard deviation 1.5), the hardness at the initial stage, to 117.9 HV (standard deviation 2.3) after soaking for 4 days. The artificial aging process of AA6061 in T4 to T6 temper occurs at 177°C in 10 h. Thus, by soaking the T4 specimens continuously at 150°C for several days, these specimens would be gradually aged to T6 temper. This hardness value subsequently maintained unchanged during further soaking to 10 days. In the case of T6 specimens, as shown in Fig. 9, after 10 days soaking in 150°C, their hardness value had negligible change.

Figures 10 and 11 show the evolution of roughness of T4 and T6 specimen during accelerated testing, respectively. The roughness of both T4 and T6 specimens on top surface and at bottom of the microchannel valleys have minor change after soaking for 10 days.

To summarize, according to these accelerated testing results, there will be no change in the roughness value of the T4 and T6 molds if they are employed in a manufacturing process with a peak cycle temperature of about 150°C. In addition, the temperature cycles of the manufacturing process will gradually harden the hardness of T4 molds to the value of T6 temper, but they will have negligible effect on the hardness of T6 molds.

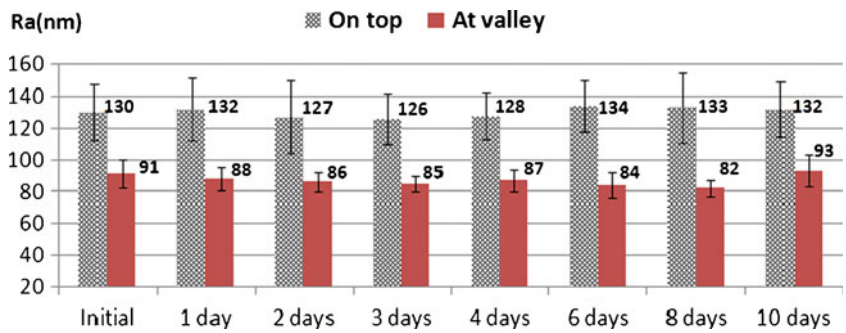

Fig. 11 Evolution of roughness of AA6061-T6 specimens during accelerated testing

4 Conclusions

In this study, the significant changes of roughness, hardness, and tensile strengths (yield stress and UTS) of AA6061 mold after hot embossing, after T4 and after T6 tempering were evaluated. The results show that after hot embossing, the roughness decreased on both the top surface of the specimen and at the bottom valley of the microchannels, while the hardness and the tensile strength decreased substantially. T4 tempering after hot embossing recovered partially the hardness and tensile strengths of AA6061 specimens but with a corresponding increase in roughness. Full recovery of strengths and hardness was achieved by T6 tempering, but with a rougher (and still well acceptable) surface than T4 tempering. In addition, accelerated testing indicates that the temperature cycles in the subsequent manufacturing process of polymeric microdevices will have no effect on the roughness value of both T4 and T6 molds. Moreover, this process will improve the hardness of T4 mold to T6 temper, while the hardness of T6 molds will remain unchanged. Consequently, depending on specific requirements, trade-offs between surface roughness to strengths and hardness can be achieved by different post-tempering after the hot embossing process.

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