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Thermoanalytical characterization of thermoset polymers for chemical mechanical polishing☆

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Abstract

Thermal analytical study of two types of polyurethane-based polishing pads for chemical mechanical planarization (CMP) was conducted using DMA, TMDSC, TMA, and TGA. The pads were subjected to thermal treatments at various temperatures for different time. Based on the results of thermal analysis, recommendations to optimize pad properties were made.

Keywords: Chemical mechanical planarization; Thermal analysis; Pad

1. Introduction

Chemical mechanical planarization (CMP) has become a method of choice for planarization of metal and oxide layers in the microelectronics industry [1]. A typical CMP process consists of polishing of the metal or oxide layer using a polymer-based pad and a slurry containing abrasives. Although CMP appears to be the only viable technology for planarization of silicon wafer layers, the fundamental mechanisms involved in CMP are not fully understood [2].

During CMP process, a pad can be subjected to high temperature as a result of mechanical friction between a pad and a silicon wafer in the solid–solid contact mode [3]. This heating effect is partially alleviated in the hydro-dynamical contact mode [4] due to the slurry flow. It was found empirically that the slurry temperature could increase by approximately 20–30 °C during CMP. However, these data reflect the averaged temperature over the pad/wafer contact, while purely elastic model (that should be valid for soft polymer-based pads [3]), predicts that only about 1% of the pad surface is in contact with the wafer during CMP [1]. As such, the local pad temperature during CMP can increase significantly, especially at the points of contacts between the trench edges and the pad. Pad heating can substantially and irreversibly change the physical and mechanical properties of the pads [5–8] and their chemical structure. Temperature effect on the pad properties depends on the pad type and design; there are various CMP pads used for different CMP applications. This study deals with the effect of pad thermal conditioning on the thermal and mechanical properties of two types of polyurethane-based pads.

2. Experimental

2.1. Materials

Two types of circular polyurethane pads were employed in this study, namely, a concentrically grooved one-layer micro-porous pad, and a multi-layer embossed pad containing both micro- and macro-pores. The pads differ by their design, manufacturing process, and hardness. The one-layer and multi-layer pads were referred as a “hard” pad and a “soft” pad, respectively. The pad samples were conditioned in the oven at 70, 110, 150, and 190 °C for 1 h, and at 110 °C for 1, 2, 4, 8, and 24 h. The adhesive layers used to maintain a pad on the polishing platens were removed from the pad prior

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to the conditioning and subsequent testing. For DMA testing, specimens were cut from the circular pads close to the pad edges. For hard pads, specimens with longitudinal groove orientation with respect to the long side of the rectangular specimen were tested.

2.2. Characterization

Pad samples were characterized using dynamic mechanical analysis (DMA), thermal modulated differential scanning calorimetry (TMDSC), thermal gravimetric analysis (TGA), and thermal mechanical analysis (TMA). The set of four analytical tools was manufactured by TA Instruments, Inc., and operated using a TA Instruments Thermal Advantage operating system. Prior to testing, the thermally conditioned samples were stored in a desiccator.

2.2.1. DMA

Dynamic mechanical tests were performed using a DMA 2980 in the single cantilever bending mode for the "hard" pads and in tensile mode for the "soft" pads. A support frame with a span of 41 mm was used in the bending mode. The samples were held in the DMA fixture clamps using a torque value of 110 N cm. After mounting and prior to the testing, the specimens were kept on the support frame for approximately 10 min to release stresses possibly introduced during mounting. The 218 mm × 13 mm × 2 mm specimens were tested using a multi-frequency deformation mode at frequencies of 1, 10, and 100 Hz, a heating rate of 5 °C/min, and oscillating amplitude of 30 μm. The typical temperature range for the tested specimens was from −120 to 175 °C. Liquid nitrogen was used for sub-ambient testing. Dry nitrogen was used to provide support for DMA air bearing; the excess of nitrogen was released in the DMA oven and served as a purged gas in the DMA tests. In a typical DMA run, the following parameters were determined: dynamic storage flexural, G', or tensile, E', moduli in the whole temperature range, area under and the peak of the damping curve, tan δ, and the temperature at the peak of dynamic loss modulus, G'', or glass-transition temperature.

2.2.2. TMA

Thermomechanical testing was performed using a TMA 2940. An expansion macroprobe and a penetration microprobe were used in the study. The probe loads were selected at 0.200 and 0.100 N for macroprobe and microprobe, respectively. The approximately 7 mm × 7 mm specimens were placed in the TMA cell in such a way that the grooved, or embossed surfaces for "hard" and "soft" pads, respectively, were in contact with the probe, and the back surface was in contact with the hot plate. The samples were heated in the temperature range from either ambient temperature, or −120 °C, to 200 °C at a heat rate of 5 °C/min. Dry nitrogen at a rate of 30 mL/min was used as a purged gas. Liquid nitrogen was used to achieve sub-ambient temperatures.

2.2.3. TMDSC

Modulated differential scanning calorimetry was performed using an MDSC 2910 from TA Instruments, Inc., while purging with dry nitrogen at a flow rate of 10 mL/min. The weight of each specimen was between 5 and 10 mg. The scanning rate was 3 °C/min in the temperature range from −40 to 200 °C. Temperature oscillating amplitude and period were ±1 °C/min and 30 s, respectively. The samples were first heated in the specified range (first heat), cooled down to −40 °C, and heated again till the maximum 200 °C (second heat). The calorimeter was calibrated at various temperatures using standard procedures and calibration metals. The temperature dependencies of heat capacity, and a regular, non-reversing, and reversing heats were received in one run in TMDSC. In a typical TMDSC run, endo- or exothermic heat associated with a chemical reaction or a phase transition, were determined as an area under the heat peak, and glass transition temperature was determined as the midpoint of the shift on the heat-temperature curve.

2.2.4. TGA

Thermogravimetric tests were performed using a TGA. A sample of approximately 10 mg was placed into the crucible, and heated from the room temperature to 350 °C at a heat rate of 10 °C/min. The weight loss as a function of temperature was recorded during the test.

3. Results and discussion

3.1. DMA analysis

The typical DMA runs for the hard and soft pads conditioned at room temperature and tested at a frequency of 100 Hz are shown in Figs. 1 and 2. Storage moduli, G' and E', and tan δ were determined over the wide range of temperatures. Loss moduli, G'' and E'', were the product of $G'(E')$

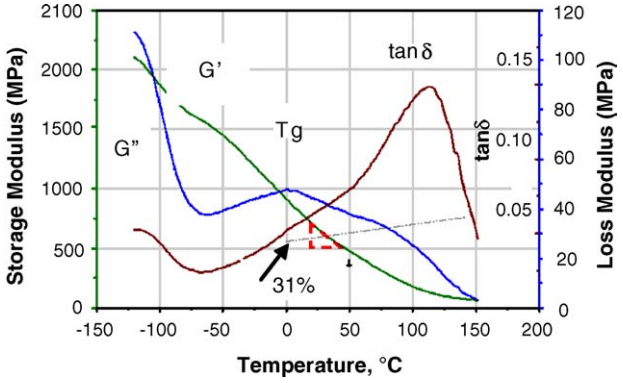

Fig. 1. DMA scan for as-manufactured pad, tested at a frequency of 100 Hz. Decrease of 31% in G' was observed in the temperature range from 25 to 50 °C.

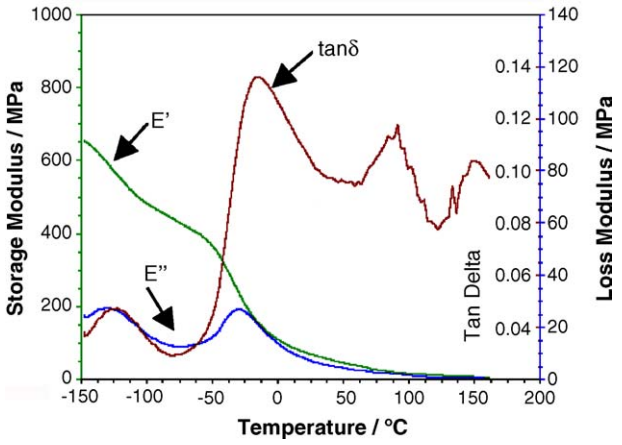

Fig. 2. DMA scan for soft pad conditioned at RT and tested at a frequency of 100 Hz. Decrease of 50% in E' was observed between 25 and 50 °C.

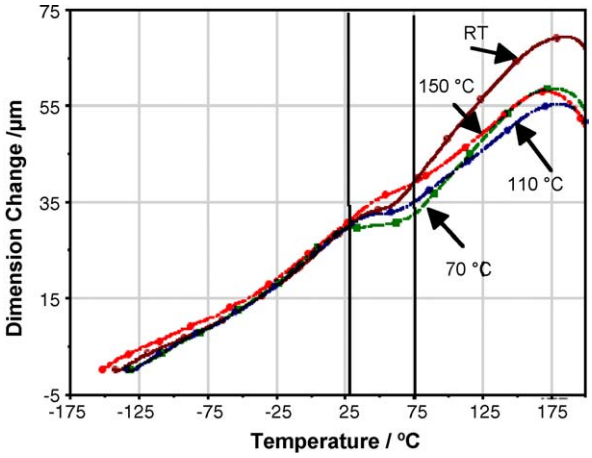

Fig. 3. TMA runs for the hard pad. Three distinguished areas characterized by different CTE are observed.

and tan δ. Moduli G' and E' decreased by an order of magnitude as temperature increased from −120 to 150 °C, as shown in Figs. 1 and 2. In the typical for CMP process temperature range between 25 and 50 °C moduli G' and E' decreased by 31 and 50%, respectively, as shown in Figs. 1 and 2. Decrease of modulus results in the change of pad hardness and, as such, pad polishing performance [5–8]. An ideal polishing pad should show no change of the storage modulus within the operating temperature range.

Glass transition temperature was assigned as the peak of storage moduli [9], G'' and E'', and was observed at approximately 0 and −30 °C for the hard and soft pad, respectively, as shown in Figs. 1 and 2. Damping curve, tan δ, is associated with the partial loosening of the polymer structure so that smaller chain segments become more mobile [9]. For most polymers, the temperature at the tan δ peak is approximately 15–20 °C higher than the temperature at the G'' peak [10]. However, unusually high difference of more than 100 °C between these two temperatures was observed for the hard pad.

The height of the damping peak is a measure of mobility of the molecular chains [9], and relates to the degree and strength of chemical bonds in the polymer structure. In this study, it was used to determine the effect of temperature conditioning on the polymer structure.

3.2. TMA analysis

The typical TMA run for the hard pad conditioned at room temperature and tested using a penetration microprobe is shown in Fig. 3. Three distinguished areas that show different coefficients of thermal expansion, CTE, were observed: (1) below approximately 25 °C, CTE = 72 µm/m°C; (2) between 25 and approximately 50 °C, CTE = 0 µm/m°C; (3) above 50 °C, CTE = 146 µm/m°C. The typical temperature operating region of a CMP process varies between room temperature and approximately 50 °C. As such, CTE of the tested pad did not change within the typical operating region. However, if temperature on the pad changes by approximately 10 °C above 50 °C or below 25 °C, the CTE increases dramatically to 72 µm/m°C or 146 µm/m°C, respectively. As a result, pressure applied to the wafer increases and in turn, CMP polishing performance will change due to the change in CTE. It is therefore important to use pads with a stable CTE within the CMP temperature operating range.

For the soft pads, TMA results were obtained using a macro probe. Four ranges characterized by different CTE, were observed for soft pads as shown in Fig. 4. Below approximately 30 °C where CTE was steadily increasing; between approximately 30 and 50 °C, characterized by relatively stable CTE; between approximately 50 and 85 °C, where CTE was steadily decreasing; and above 85 °C characterized by sharp drop of CTE. For soft pads, the temperature above 85 °C should be avoided.

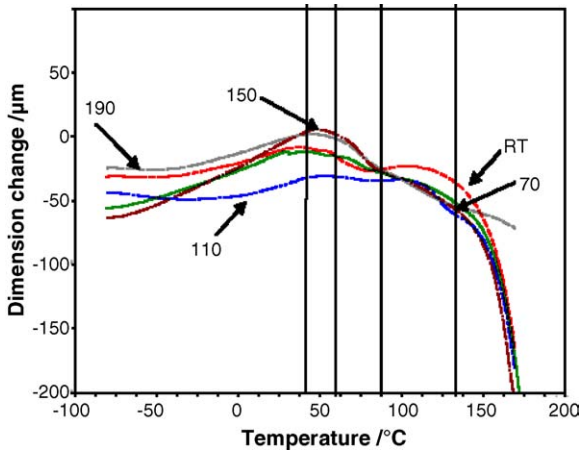

Fig. 4. TMA runs for the soft pad. Four distinguished areas characterized by different CTE are observed.

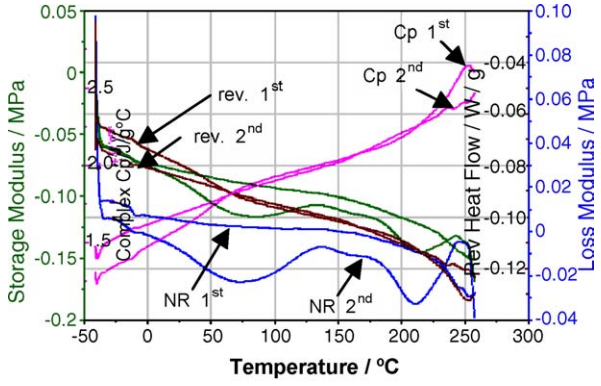

Fig. 5. TMDSC run as-manufactured hard pad. Two NR endothermic and one NR exothermic peak are observed in the first heat run. All peaks disappeared in the second heat run. NR, rev, and C_p stand for non-reversing heat, reversing heat, and heat capacity; 1st and 2nd stand for the first and second heats, respectively.

3.3. DSC analysis

Modulated DSC runs for conditioned at room temperatures hard and soft pads are shown in Figs. 5 and 6, respectively. Reversing heat and heat capacity did not show any meaningful peaks as shown in figures. Small peak at around 0 °C are present in DSC runs for both hard and soft pads, and are probably an artifact related to ice/water transition. Non-reversing (NR) heat curves for hard pads show the following peaks: (1) endothermic peaks located in the ranges between 25 °C and 130 °C, (2) onset of another endothermic peak at 180 °C, and (3) an exothermic peak characterized by maximal temperature of approximately 250 °C. The second endothermic peak and the onset of exothermic peak overlaps. Non-reversing curves for soft pads show a single endothermic peak located between 25 and 125 °C. The exothermic peak, observed for the hard pads between 220 and 250 °C is ascribed to the hard pad decomposition, and confirmed by the results from TGA analysis (Fig. 7). A non-reversing exo-peak, related to decomposition, is not observed for the soft

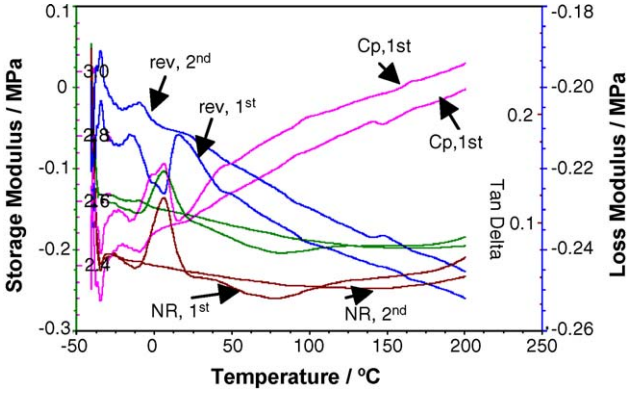

Fig. 6. TMDSC run for conditioned at RT soft pad. Only one NR endothermic is observed. NR, rev, and C_p stand for non-reversing heat, reversing heat, and heat capacity; 1st and 2nd stand for the first and second heats, respectively.

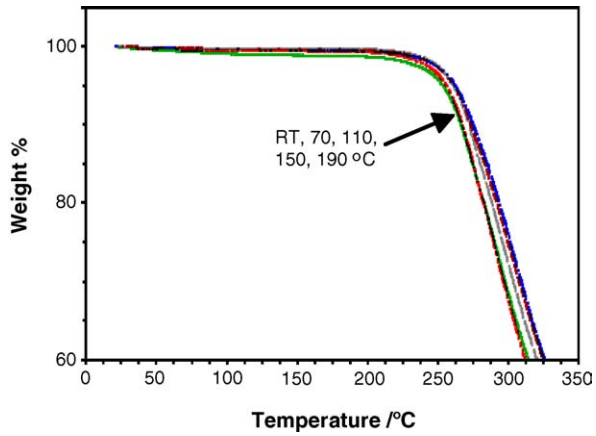

Fig. 7. TGA runs for the hard pads. No effect of conditioning on decomposition temperature is observed.

pads; one of the possible explanation is that soft pad comprises of a multi-layer stack of the polymers. In this case, expected thermal decomposition from the top layer can be shielded by thermal contributions from other layers.

The second heat runs show flat non-reversing curves for both hard and soft pads; this confirms that the observed endothermic processes in the range between 25 and 125 °C, as well as hard pad decomposition, are non-reversible processes. This endothermic non-reversible activity observed upon heating for both hard (two peaks) and soft (one peak) pads can be caused by a breakup of the crystalline regions by depolymerization, as it was observed elsewhere for thermoplastic polyurethane ETPU [11].

3.4. TGA analysis

TGA analysis shows that both hard and soft pads are characterized by only one degradation transition; degradation temperature was observed at approximately 250–260 °C, as shown in Figs. 7 and 8.

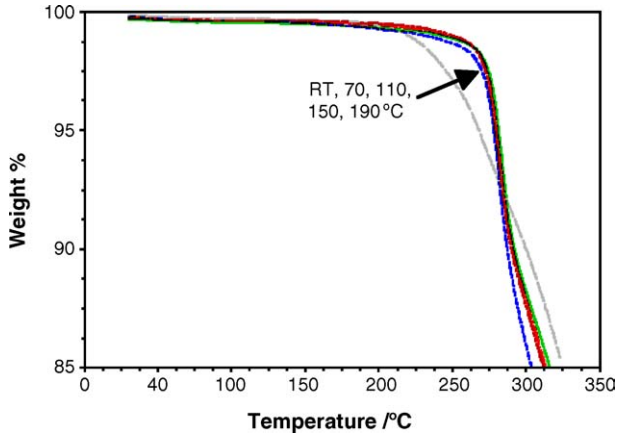

Fig. 8. TGA runs for the soft pads. No effect of conditioning on decomposition temperature is observed.

3.5. Effect of thermal conditioning on the pad properties

The effect of conditioning at various temperatures for 1 h and at a temperature of 110 °C for different conditioning time was studied. The temperature of 110 °C was selected since it was well below the onset of decomposition of both hard and soft pads determined using TGA analysis. The following parameters were monitored for DMA tests: percentage of decrease of storage modulus measured at 25 and 50 °C; glass transition temperature; and macromolecular mobility. The following parameters were monitored for TMA tests of the hard pads: CTEs measured at the temperatures below 25 and above 50 °C; low and high limit temperatures, shown as T_{low} and T_{high} in Fig. 2; and the temperature range, $T_{dif} = (T_{high} - T_{low})$, within which CTE was equal to approximately 0 μm/m°C. For soft pads, CTEs in four ranges, 35–60, 60–80, 80–130, and above 130 °C were monitored. For DSC and TGA tests, the effect of temperature on endothermic non-reversing heat and location of decomposition temperature were monitored.

3.5.1. Effect of temperature

The specimens were conditioned at 70, 110, 150, and 190 °C for 1 h. Additionally, original, as manufactured specimens, conditioned at room temperature, were tested. Thermal decomposition was visually evident for the specimens conditioned at 190 °C. As such, the test results for these specimens were shown on the plots, but were excluded from the analysis and discussion.

3.5.1.1. DMA tests. For hard pads, temperature conditioning affected percentage of reduction of the storage modulus within the operating temperature range between 25 and 50 °C, as shown in Fig. 9. Percentage of reduction, PR, was calculated as

$$\mathrm{PR}_{\mathrm{shear}}\,(\%) = 100 \times \left(\frac{G'@25\,^{\circ}\mathrm{C} - G'@50\,^{\circ}\mathrm{C}}{G'@25\,^{\circ}\mathrm{C}} \right) \quad (1)$$

where G'@25 °C and G'@50 °C are storage shear moduli measured at 25 and 50 °C, respectively.

Conditioning at 70 and 110 °C provided an approximate 2% reduction in the modulus decrease, as shown in Fig. 9. For the hard pad, temperature conditioning can alleviate the problem of modulus reduction within the typical operational range. The optimal annealing temperature for the hard pads was determined at 70 °C, as shown in Fig. 9. However, temperature conditioning does not substantially affect percentage of the change of tensile storage modulus of the soft pad within the same operating temperature range between 25 and 50 °C, as shown in Fig. 10. Percentage of reduction of tensile storage modulus for the soft pads was calculated as

$$\mathrm{PR}_{\mathrm{tensile}}\,(\%) = 100 \times \left(\frac{E'@25\,^{\circ}\mathrm{C} - E'@50\,^{\circ}\mathrm{C}}{E'@25\,^{\circ}\mathrm{C}} \right) \quad (2)$$

where E'@25 °C and E'@50 °C are storage tensile moduli measured at 25 and 50 °C, respectively.

Similarly, temperature conditioning of hard pads affected macromolecular mobility; the heights of the damping peaks increased by approximately 2% upon heating up to 70 °C, and decreased by approximately 3–5% upon further temperature increase from 70 to 110 and 150 °C, as shown in Fig. 11. Decrease of mobility implies that additional chemical bonding, or more stable structure, is forming at elevated conditioning temperatures. The mobility results can be correlated to the measurements of NR heat in DSC tests shown in Fig. 5. Maximum non-reversing activity is observed at approximately 70 °C, at which point crystallite regions break up, and mobility of macromolecular chains increase. At 110 and 150 °C, the process of breakup mostly completes, and movement of macromolecular chains become more restricted due to other factors that yet to be understood. For the soft pads, the opposite effect of temperature on mobility was observed.

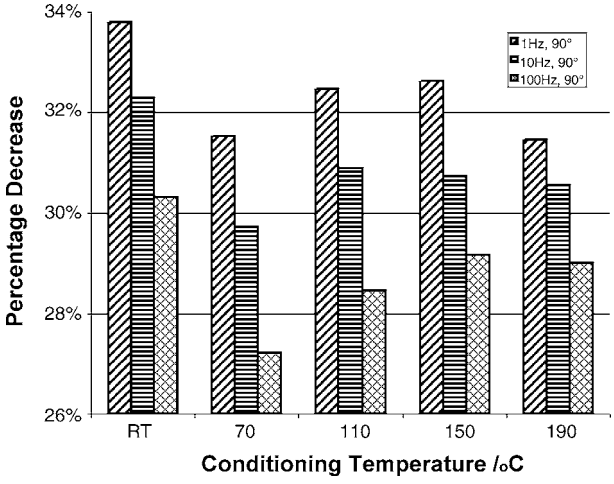

Fig. 9. Effect of temperature on percentage of reduction of storage shear modulus G' in the typical operating temperature range (25–50 °C). Results are shown at three DMA test frequencies. Thermal conditioning reduces percentage of modulus reduction.

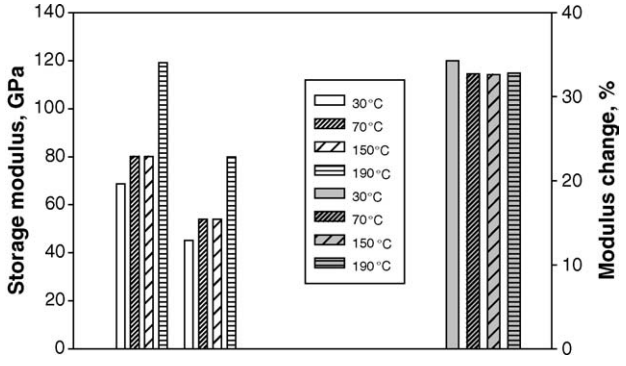

Fig. 10. Effect of temperature on storage tensile moduli and percentage of modulus reduction in the typical operating temperature range (25–50 °C). All results are shown for one DMA test frequency, 10 Hz. Thermal conditioning does not substantially affect percentage of modulus reduction.

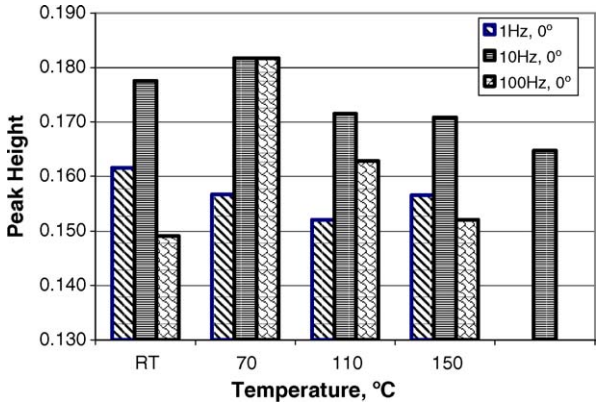

Fig. 11. Effect of temperature on macromolecular mobility of the hard pads. Mobility is measured as the heights of the damping peaks, tan δ. All results are shown for three DMA test frequencies. Thermal conditioning increase mobility at 70 °C, and decrease it upon heating above 70 °C.

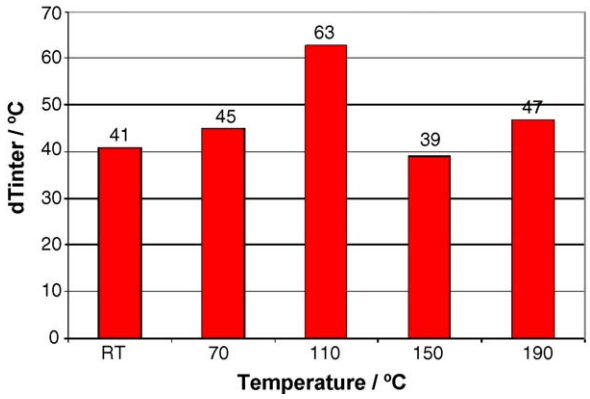

Fig. 13. Effect of temperature on width of the temperature range within which CTE of the hard pads is stable (close to zero). Conditioning at 110 °C for 1 h provided the widest temperature range of stable CTE.

As shown in Fig. 12, mobility increased as conditioning temperature increased. For the soft pads, mobility increase can be tentatively explained by the dominant effect of breakup of crystalline regions upon heating, which leads to mobility increase. Multiple damping peaks, shown in Fig. 12, are the results of the contribution of the multiple polymer layers in the pad stack.

3.5.1.2. TMA tests. The effect of conditioning temperature on the width of the temperature range T_{dif} was studied. The widest T_{dif} of 63 °C (from 11 to 74 °C), was observed for the pad conditioned at 110 °C as shown in Fig. 13. The second widest T_{dif} of 45 °C (from 42 to 87 °C) was observed for the pad conditioned at 70 °C. As such, proper temperature conditioning allows expansion and shift of the operating range for the hard pads. For the soft pads, temperature conditioning did not substantially affect the temperature limits of four regions indicated in Fig. 4. As such, temperature conditioning cannot be used to optimize performance of the soft pads.

3.5.1.3. DSC. Temperature conditioning of the hard pad decrease NR endothermic peak as shown in Figs. 14 and 15. This phenomenon can be explained by the breakup of crystallite regions. Conditioning at 110 and 150 °C practically eliminates any thermal pre-history of the hard pads by breaking up crystalline regions in the pads. Conversely, temperature conditioning of the soft pads did not affect NR endothermic heat as shown in Fig. 16. Apparent stability of crystalline regions in the soft pads can be an artifact due to the contributions of the competing effects from the bottom layers of the multi-layer soft pad.

3.5.1.4. TGA. No effect of temperature conditioning on decomposition temperature was observed neither for hard, nor for soft pads as shown in Figs. 7 and 8. The decomposi-

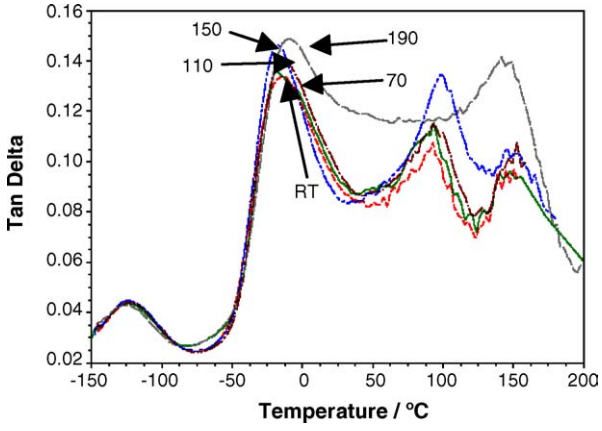

Fig. 12. Effect of temperature on macromolecular mobility of the soft pads. Mobility is assigned as the heights of the damping peaks, tan δ. The results are shown for one DMA test frequency of 10 Hz. Mobility increases as conditioning temperature increases. Thermal conditioning increase mobility. Multiple peaks reflects multi-layer structure of soft pads.

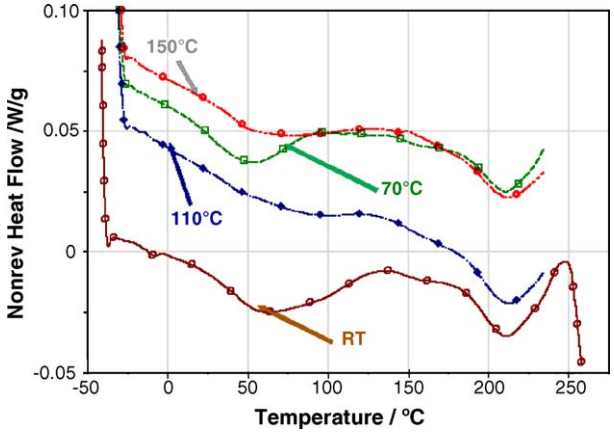

Fig. 14. Effect of temperature conditioning on non-reversing heat measured in TMDSC tests for the hard pads (qualitative data). Temperature conditioning decreases NR heat; thus, conditioning is recommended to stabilize thermal properties of hard pads.

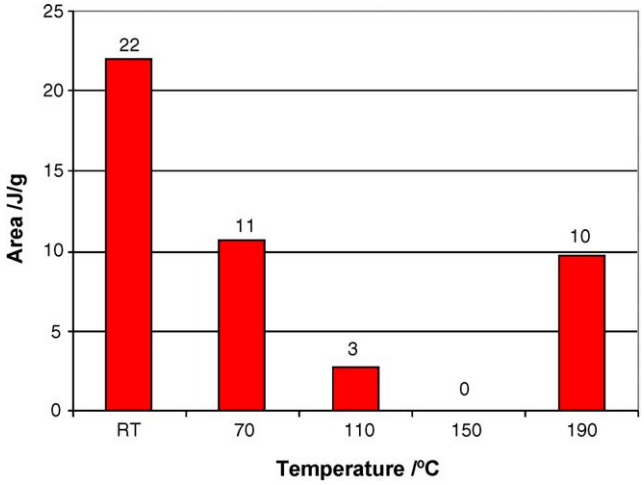

Fig. 15. Effect of temperature conditioning on non-reversing heat measured in TMDSC tests for the hard pads (quantitative data). Temperature conditioning at 110 and 150 °C eliminates thermal pre-history of hard pads.

tion temperatures of the tested pads were well above the pad operating range.

3.5.2. *Effect of time*

Since temperature conditioning of the soft pads did not substantially affect their properties, effect of conditioning time was studied for the hard pads only. The pad specimens were conditioned at 110 °C for 1, 2, 4, 8, and 24 h.

3.5.2.1. DMA. Conditioning for 1–8 h did not reduce percentage of reduction of storage modulus as shown in Fig. 17. Conditioning for 24 h provided small, 1–2% improvement in the modulus decrease, as was calculated using Eq. (1). As such, conditioning time has lesser impact on reduction of storage modulus, than conditioning temperature.

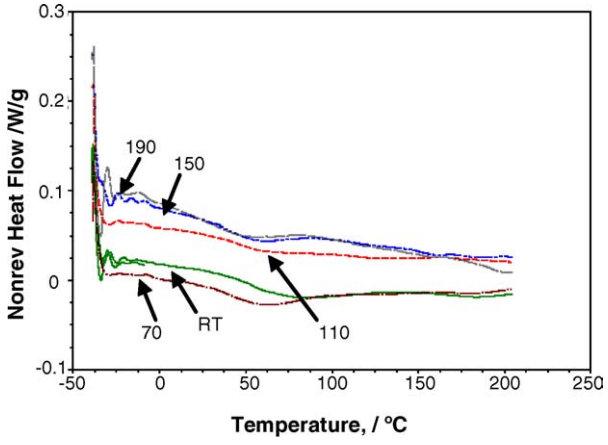

Fig. 16. Effect of temperature conditioning on non-reversing heat measured in TMDSC tests for the soft pads (qualitative data). Temperature conditioning does not substantially affect NR heat (NR heat were similar for conditioning at different temperatures).

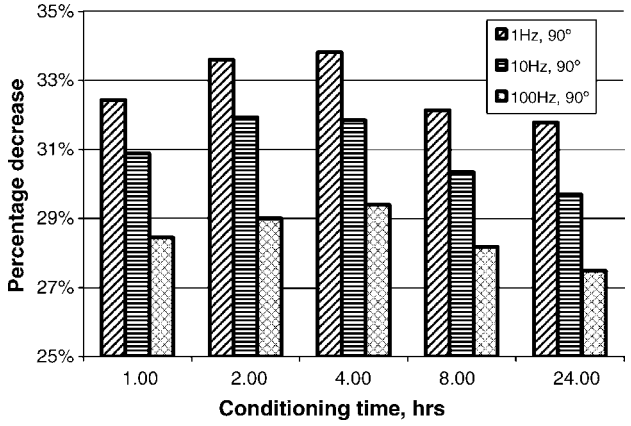

Fig. 17. Hard pads: effect of conditioning time at 110 °C on percentage of reduction of shear modulus within the temperature range of 25–50 °C. The results are measured at three DMA test frequencies. Conditioning for 8–24 h reduces percentage change by 1–2%.

Similarly, conditioning for 1–8 h did not significantly affect mobility of macromolecular chains, while conditioning for 24 h decreased mobility by 3–6% as shown in Fig. 18.

The decrease of mobility with increase of conditioning time and temperature indicates that the original pad was not completely thermally stabilized; conditioning at elevated temperatures for sufficiently long time stabilizes the pads and improves pad CMP performance. Additional temperature conditioning may be a necessary step to optimize properties of as-manufactured polyurethane-based pads.

3.5.2.2. TMA. The widest T_{dif} was observed for the pad conditioned for 8 h as shown in Fig. 19. As such, long temperature conditioning can be beneficial for hard pad performance.

3.5.2.3. DSC, TGA. Effect of time of temperature conditioning on endothermic NR heat was not pronounced, if any. Conditioning time did not affect decomposition temperature.

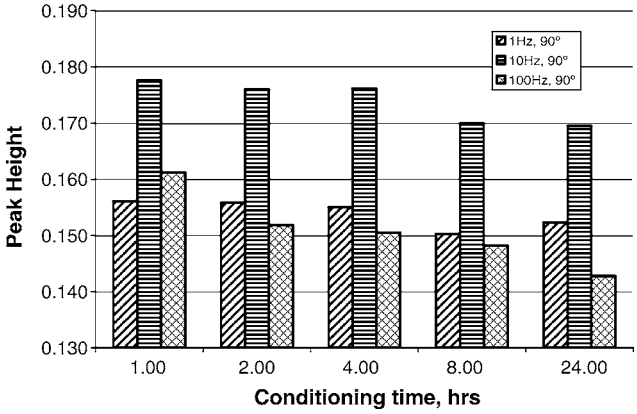

Fig. 18. Hard pads: effect of conditioning time at 110 °C on mobility measured as the heights of the damping peaks. The results are measured at three DMA test frequencies. Conditioning for 8–24 h reduces mobility by 3–6%.

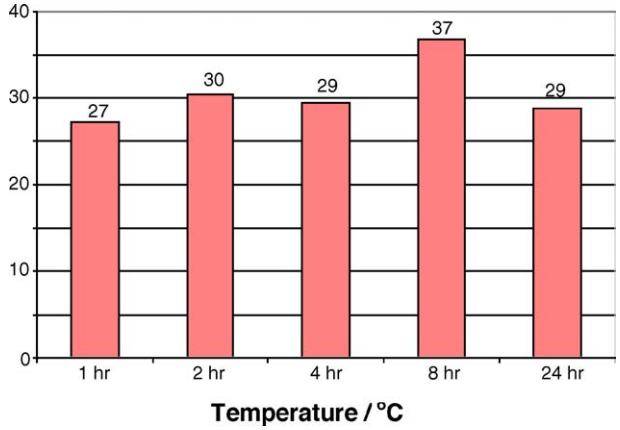

Fig. 19. Hard pads: effect of conditioning time at 110 °C on width of the temperature range within which CTE is stable. The results are measured at three DMA test frequencies. Conditioning for 8 h provides the widest temperature interval of the stable CTE.

Overall, conditioning of the hard pads at 110 °C for 8–24 h provided the most significant improvement of the properties of the original, as-manufactured hard pads.

4. Conclusions

1. Change of approximately 30% in the value of the pad storage modulus within the typical temperature range of a CMP process was demonstrated. For the hard pads, conditioning at elevated temperatures decreases this change.
2. Existence of three and four temperature ranges, characterized by different coefficients of thermal expansion, CTE, was established for hard and soft pads, respectively. Only hard pad show stable CTE in the typical temperature operating range.
3. Optimal temperature operating range for CMP polishing pads was established.
4. Temperature conditioning improves properties of the original, as-manufactured polyurethane-based hard pads, but does not significantly affect properties of the soft pads.
5. Optimal temperature and time of the pad post-manufacture conditioning were recommended for the hard pads.

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