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Research Article

The Effect of Strain Rate on the Tensile Properties of PMMA/Hydroxyapatite Composite

M. Farjamfar[1], S.M. Zebarjad[1*], M.E. Bahrololoom[1] and R. Bazargan-Lari[2]

[1] Department of Materials Science and Engineering, School of Engineering, Shiraz University, Shiraz, Iran
[2] Department of Materials Science and Engineering, Marvdasht Branch, Islamic Azad University, Marvdasht, Iran

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ABSTRACT

To investigate the effect of the different content of hydroxyapatite (HA) (5, 10, and 15 wt.%) on the tensile properties and deformation mechanism of the poly methylmethacrylate (PMMA), tensile test at different strain rate (4.1×10^{-4}, 8.3×10^{-4}, 1.66×10^{-3}, 3.31×10^{-3}, 8.275×10^{-3}, and 16.6×10^{-3} s^{-1}), microscopic evaluations were performed on the standard samples. The results of the tensile test showed that by adding HA to the PMMA, its strain rate sensitivity increased. The activation energy (ΔH) for chain fluctuation decreased from 56.42 to 37.81 J/mol as HA content increased from 0 to 15 wt.%. In addition, the active volume (V^*) of the chain during the tensile test was measured indicating that its value decreased from 3.02 to 2.35 as HA content promoted from 0 to 15 wt.%. The results of microscopic evaluation showed that the deformation mechanism of PMMA during the tensile test was crazing and dispersity of craze depended strongly on the HA content.

1. Introduction

Polymethylmethacrylate (PMMA) is a transparent thermoplastic material that has vast applications due to its optical clarity, high impact resistance, and ease of fabrication. PMMA is commonly used in the construction industry for its durability and resistance to weathering. It can be used in windows, skylights, and domes, as well as in lighting fixtures and signage. PMMA is also used in the automotive industry for its optical clarity and shatter resistance, and in the medical industry for its biocompatibility and transparency [1]. It is also cost effective, stronger than the bone adjacent to the skull in mechanical testing and inert with minimal inflammatory reaction [2]. These properties make it very useful in various fields such as bone cement for fixing orthopedic implants to the skeleton, as contact lenses and intraocular, dental uses and so on [3-6]. Although PMMA has unique features such as its ease and aesthetic

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[*] Corresponding author
E-mail address: mojtabazebarjad@shirazu.ac.ir (S.M. Zebarjad)

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fabrication, low cost, and favorable physical and mechanical properties, nonetheless, these are insufficient in providing the necessary wear resistance, hardness, and mechanical strength. In order to promote its properties adding additional fillers and reinforcements to it is very common [7-10]. In fact, unmodified (PMMA) has brittle behavior, especially under an impact force and is also particularly susceptible to scratching in comparison with conventional inorganic glasses. Due to the limited mechanical properties of this polymer and its weak compatibility with bones, its medical usage is sometimes associated with complications. In order to improve these substantial deficiencies, a vast number of research studies have been done on polymer matrix composites. In fact, adding a bioactive minerals agent such as Sb_2O_3, Al_2O_3, TiO_2, and hydroxyapatite (HA) particles can improve the mechanical and biocompatibility properties of organic polymers [2, 8, 9]. Because of the similar structure of hydroxyapatite to bone and its good osteogenesis ability, it can be an excellent choice as a component of biocompatible composite for medical applications [6]. However, depending on the HA content, the mechanical and thermal properties of the composite can be different [3]. Indeed, HA is inherently brittle but has good osteoconductivity, and PMMA has a higher fracture resistance and easy processing but has relatively weak osteoconductivity. This has caused the most common composite, the so-called HA-PMMA, used in cranioplasty [3]. Based on our knowledge there are some papers that have concentrated on the PMMA/HA composites. For example, Wijesingh et al. [11] synthesized hydroxyapatite/polymethylmethacrylate (HA-PMMA) nanocomposites using an economical, simple, and industrially applicable novel method by utilizing extensively distributed dolomite. Prahasanti et al. [12] reported there is a possibility of utilization of PMMA/HA composite as a biomaterial candidate for porous trabecular dental implant fixtures. Aldabib and Mohd Ishak. [13] reported that adding HA to PMMA caused an increase in its flexural strength [13]. Karla [14] presented an article focused on the role of HA in the compression behavior of PMMA. According to the results, PMMA/HA nanocomposite exhibited an acceptable elastic modulus for its possible consideration in tissue engineering. Silva et al. [15] investigated the behavior of the PMMA/HA under gamma radiation. Ataei-Azam et al. [16] published a paper concentrated on the experimental and numerical analysis of the fracture behaviors of PMMA/HA nanocomposites under pure mode I, pure mode II, and mixed-mode (I/II) [16]. Nabha et al. [2] focused on using a hybrid filler of Al_2O_3 and TiO_2 nanoparticles with various loading content to evaluate and minimize the wear rate of PMMA [2].

The effect of both temperature and strain rate on the mechanical properties of PMMA composite has been investigated by many investigators. For example, Wu and his colleagues [17] studied the tensile properties of PMMA and showed that the mechanical properties of PMMA are very sensitive to strain rates. They showed that when the strain rate increased, both strength and modulus increased, while the fracture strain decreased [17]. The Effect of powder morphology on the tensile properties of polymer-HA composite has been studied by Cheang [18]. Zebarjad et al. [19] showed that the presence of the needle-shaped HA affected the tensile properties of PMMA. Based on a literature survey done by the authors, it seems that the role of both strain rate and HA content on the deformation mechanism of PMMA has not been under attention and the authors try to fill the literature gap in this issue, and the present study has been conducted on this basis.

2. Experimental Procedure

2.1. Sample preparation

Heat-cure acrylic powders (Acropars, Marlic) and methyl methacrylate (Acropars, Marlic) were used to produce polymethylmethacrylate (PMMA) as polymeric composite. Hydroxyapatite powders (extracted from bovine cortical bone ash) were used as a reinforcement phase. Indeed, hydroxyapatite powder was prepared by burning bone and heat-treating the bone ash at 800-850°C in an air furnace for 3 h. The block ash was converted to a white powder after heat treatment. Afterward, the powder was crushed by a ball mill at a constant ball/powder ratio of 1:2 for 2 h.

To prepare composite samples, weighed PMMA and HA powders were mixed by mechanical milling for 30 min. The weight ratio of balls to powders was kept constant at 1:2 (20 g balls and 40 g powders). Finally, PMMA composites reinforced with different content of HA (5, 10, and 15 wt.%) were produced. To fabricate tensile specimens, the required amount of methyl methacrylate monomer was poured into a clean container and almost 3 times of heat-cure acrylic powders were added to it and stirred by spatula for 30 s and the container was tightly closed. Nearly 6 min after mixing the powder and liquid at room temperature, the mixture came to dough. Then the dough was poured into the mold and pressed for 20 min. Afterward, the mold was boiled in water for 20 min.

2.2. Characterization

X-ray diffraction (XRD) patterns were recorded on a Bruker D8 Advance X-Ray diffractometer with a Cu K_α anode (λ=0.154 nm) operating at 40 kV and 40 mA. The diffraction patterns were collected at 25°C and over an angular range of 5° to 100° with a scan rate of 0.05°/min. The morphology of produced HA was investigated using scanning electron microscopy (SEM, S 320, Cambridge). To avoid accumulation of negative charge over the surface of HA powders the samples were deposited by gold in sputtering chambers with a low pressure of about 10^{-2} MPa. Tensile test was performed in accordance with ASTM D638 at different cross-head speeds of 0.25, 0.5, 1, 2, 5, and 10 mm/min corresponds to 4.1×10^{-4}, 8.3×10^{-4}, 1.66×10^{-3}, 3.31×10^{-3}, 8.275×10^{-3} and 16.6×10^{-3} s^{-1}, respectively. For tensile testing, a computer controlled SANTAM mechanical testing machine was used. For each run 5 samples were tested, and the average values were reported.

To investigate the effect of adding HA to polymethylmethacrylate on the deformation mechanism, a tensile test with a strain rate of 8.3×10^{-4} s^{-1} was performed on the PMMA samples and their composites. The morphology of produced HA was evaluated using SEM (SEM, S 320, Cambridge). In order to find out the role of HA on the deformation mechanism of PMMA, the side surface of the specimen was polished to achieve a surface roughness of about 4 µm. The side surface of specimens before tested and in the moments the stress was equal to 0.25, 0.5, and 0.75 yield stress was observed by optical microscopy.

3. Results and Discussion

3.1. Materials characterization

Fig. 1 shows the XRD spectrum taken from the burned and calcinated cow bone powder. As seen, the spectrum corresponds to HA. The XRD pattern of hydroxyapatite typically shows characteristic diffraction peaks at 2θ values of 25.9°, 28.4°, 32°, 32.2°, 39.8°, 46.6°, and 49.5°. The (002) peak at 2θ=25.9° is the most intense peak in the XRD pattern, followed by the (211) peak at 2θ=28.4°. The (300) peak at 2θ=32° is also a prominent peak in the HA XRD pattern.

SEM micrograph shows the average particle size of the powder after 2 h grinding is about 500 nm (Fig. 2).

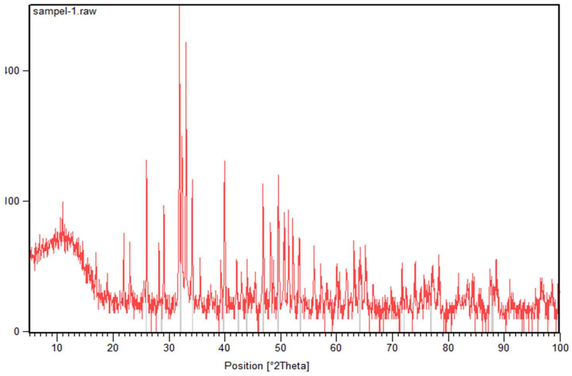

Fig. 1. X-ray diffraction pattern of produced pure HA.

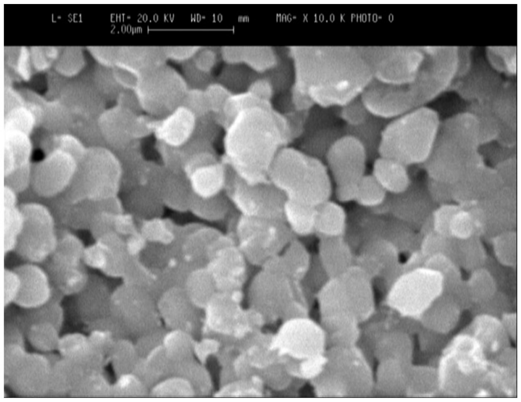

Fig. 2. SEM micrograph taken from the produced hydroxyapatite powder.

3.2. Tensile behavior

Fig. 3 shows stress-strain curves for pure PMMA at different strain rates. As seen, tensile strength increases with increasing strain rate. The reason for the dependency of tensile strength on strain rate is that, at high rate of strain, PMMA molecular chains do not have enough time to do their own makeup in the direction of stress. In fact, because of the free volume in the PMMA polymer, each component of the chain has a limitation to change its location. This is due to the attractive and repulsive forces between that part of the chain and adjacent chains. This section can change its position in order to overcome the energy barrier between the chains. It is clear that the needed energy to overcome the energy barrier is obtained by applying an external stress. The more the strain rate of PMMA increases, the more energy will be blocked. In fact, by increasing the strain rate, PMMA chains do not have enough time to feel the stress, they lose the ability of the flow due to a decrease in stress.

The mechanical properties of the particulate filled composite is dependent on several factors including geometry of the reinforcement materials (shape, size, size distribution), particle surface morphology and structural characteristics of the particles, concentration and its concentration distribution in a polymer matrix [20]. To study the tensile properties of biocompatible PMMA/HA composites, tensile tests were performed at different strain rates on specimens with different HA content. Table 1 shows the results of the tensile test done on PMMA/HA composites.

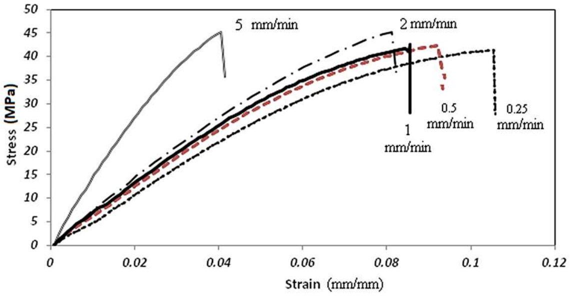

Fig. 3. Stress-strain curves for pure PMMA at different strain rates.

Table 1. Tensile properties of PMMA/HA composites

Material	**Strength (MPa)**					
Cross-head speed (mm/min)	**0.25**	**0.5**	**1**	**2**	**5**	**10**
PMMA	40.5	41.15	41.36	45.12	47.43	51.35
PMMA/HA 5	35.17	38.56	38.8	39.52	43.12	45.1
PMMA/HA 10	31.36	34.78	35.27	36.42	37.21	42.02
PMMA/HA 15	29.91	31.14	32.93	33.64	34.85	38.01

3.3. The flow model of Eyring

The flow model of Eyring provides a basis for analysis and contains the necessary elements for further development and refinement. Its aim is to correlate the effects of temperature and strain rate on flow mechanism. The fundamental idea is that a segment of a macromolecule must pass over an energy barrier in moving from one position to another position in the solid polymer [21].

Eq. (1) shows the flow model Eyring, where ΔH is the activation energy of the yielding process, T is the absolute temperature, $\dot{\varepsilon}$ is the strain rate (proportional to the cross-head speed), V^* is called the activation volume, $\dot{\varepsilon}_0$ is the pre-exponential factor and R is the universal gas constant [22]. Fig. 4 shows plots of σ_y/T against log $\dot{\varepsilon}$ for PMMA/HA composite as function of HA content. As seen in Fig. 4, the slope and intercept of all lines depend on HA content and increases in weight percent of HA, unlike the slope, causes a reduction in the intercept.

$$\frac{\sigma_y}{T} = \frac{R}{V^*}\left(\frac{\Delta H}{RT} + 2.303 \log\left(\frac{2\dot{\varepsilon}_y}{\dot{\varepsilon}_0}\right)\right) \qquad (1)$$

In fact, the increase of slope means a decrease in the V^* and implies a decrease in the ability of fluctuations

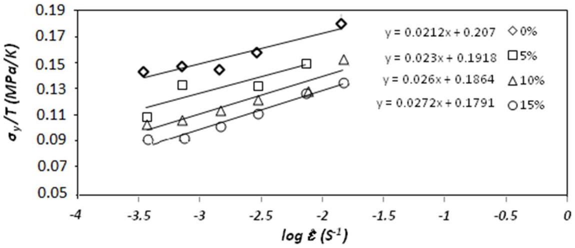

Fig. 4. Variation of σ_y/T versus log $\dot{\varepsilon}$, for PMMA/HA composites at different HA content.

of the polymer chain (Table 2). This phenomenon is due to the presence of HA particles between the polymer chains (as barriers) that impede chain movements [21]. It is true that the lack of chemical bonding between the HA particles and polymer matrix in a composite material can lead to a lower energy barrier for chains to move, especially in response to external forces. In other words, without strong interfacial bonding between the HA particles and PMMA chains, there can be still freedom of movement for the polymer chains. The presence of HA particles within a polymer matrix can indeed affect the distance between PMMA chains in the composite material. In general, the addition of HA can increase the spacing between polymer chains due to its larger size and the resulting steric hindrance effects [23]. When there is more distance between polymer chains, the intermolecular forces that hold the chains together may weaken, resulting in a reduction in the energy barrier for the chains to move or fluctuate. This can lead to an increase in the mobility and plastic deformation of the polymer chains under stress. However, it is important to note that the specific effects of the HA particles on the polymer chains and their behavior depend on many factors, including the size and shape of the HA particles, their concentration in the composite, and the interactions between the HA particles and polymer matrix. Therefore, the overall effect on the energy barrier for fluctuating PMMA chains may vary depending on the specific composition and processing conditions of the composite material. This is why σ_y/T decreases linearly with $\log \dot{\varepsilon}$ and at constant $\log \dot{\varepsilon}$, σ_y/T decreases with increasing weight percent of HA. This result is similar to that which was proposed in the previous research [22].

Figs. 5(a)-5(d) show a variation of σ versus $\log \dot{\varepsilon}$ at constant strain for the various specimens. As seen, by increasing the strain of the slope of lines constant strain rate increases. In each composite with increasing strain for a constant strain rate, the slope is increasing which reflects a decrease of fluctuations in the PMMA chains. The accident occurred due to chain stretching and compared with pure samples, the presence of HA particles decreases swing chains.

Table 2. Activation energy and activation volume of PMMA/HA composites at different HA wt.%.

Material	V^* (nm^3/segment)	ΔH (kJ/mol)
PMMA	3.02	56.42
PMMA/HA 5	2.76	47.35
PMMA/HA 10	2.45	40.57
PMMA/HA 15	2.35	37.81

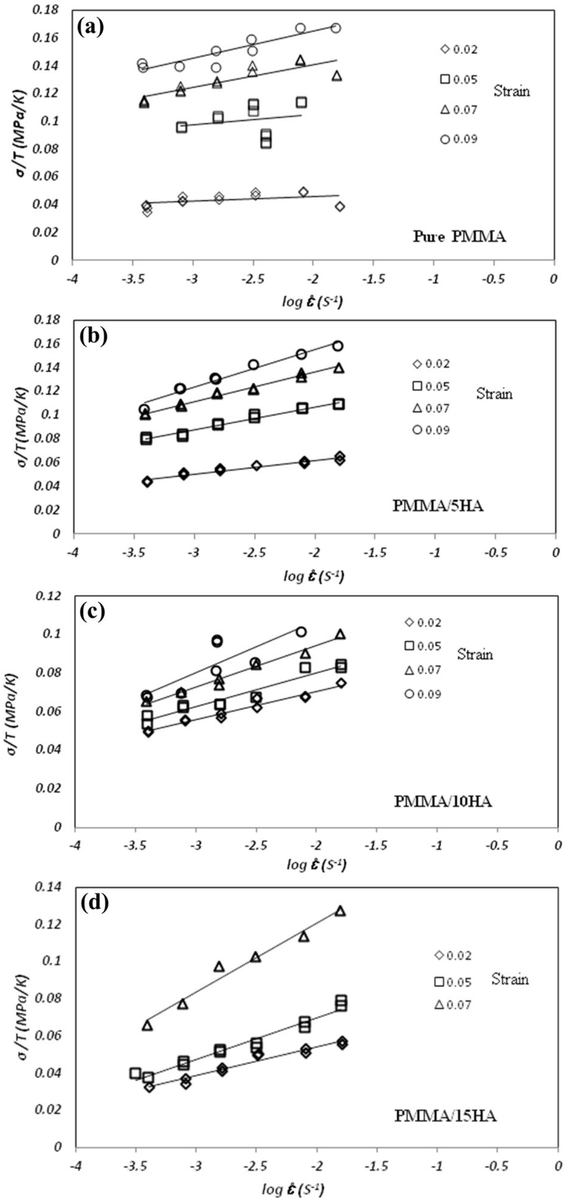

Fig. 5. The relationship between stress and $\log \dot{\varepsilon}$ at different strains.

The values of V^* and ΔH shown in Table 2 and Fig. 5 prove that an increase in the amount of HA results in a decrease in the fluctuation of polymer chain and the activation energy. At higher strains, due to a reduction in connections between adjacent chains, the activation energy is reduced. The presence of HA particles causes the connections to be reduced and it also makes the energy barrier smaller. In composites, a decrease in ΔH that is due to the increase in HA percentage and which in turn contributes to increasing the chain distances will be seen. By increasing the percentage of HA and the amount of strain because of material embrittlement, the sensitivity to strain rate is higher.

3.4. Deformation mechanism

In order to find out the deformation mechanism of PMMA and its composite, the side surface of samples was polished before testing and after applying stress the test was stopped and the polished surface was investigated using an optical microscope. Fig. 6 shows the stress-strain curve of pure PMMA tested at the strain rate of 8.3×10^{-4} s^{-1}. The positions at which tensile test were stopped to observe the side surface of sample are labeled a, b and c in the figure.

Fig. 7 shows optical micrographs taken from the polished surface of pure tensile PMMA samples tested at a strain rate of 8.3×10^{-4} s^{-1}. The samples are stretched up to the indicated stresses: (a) $\sigma = 0.25\sigma_y$, (b) $\sigma = 0. 5\sigma_y$, and (c) $\sigma = 0.75\sigma_y$. As seen, once stress increases the amount and intensity of craze zones increases and gradually the distribution of craze in the sample completely becomes uniform. In fact, crazing is a phenomenon that can occur in polymers when they are subjected to tensile loading. It refers to the formation of small, interconnected micro-cracks that appear on the surface or even in the bulk of the material. The extent and severity of crazing in a polymer material depends on several factors, including the stress value applied to the material, type of stress, stress concentrators, and the temperature or strain rate at which the test was done. At low stress values, polymers typically exhibit ductile behavior, meaning that they can deform plastically without breaking. In this regime, the formation of crazes may be limited or nonexistent, as the polymer chains can rearrange to accommodate the applied stress without undergoing significant damage [24]. However, as the stress value applied to the polymer material increases, the polymer chains may undergo greater deformation

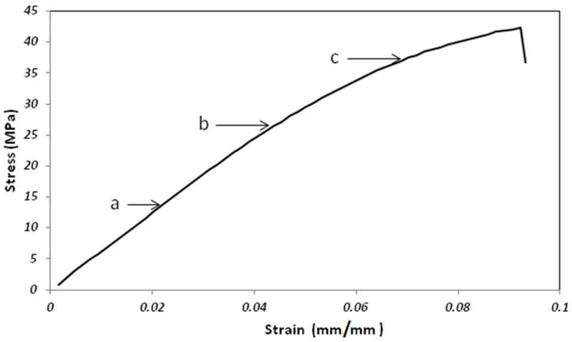

Fig. 6. Stress-strain curve of PMMA at the strain rate of 8.3×10^{-4} s^{-1}.

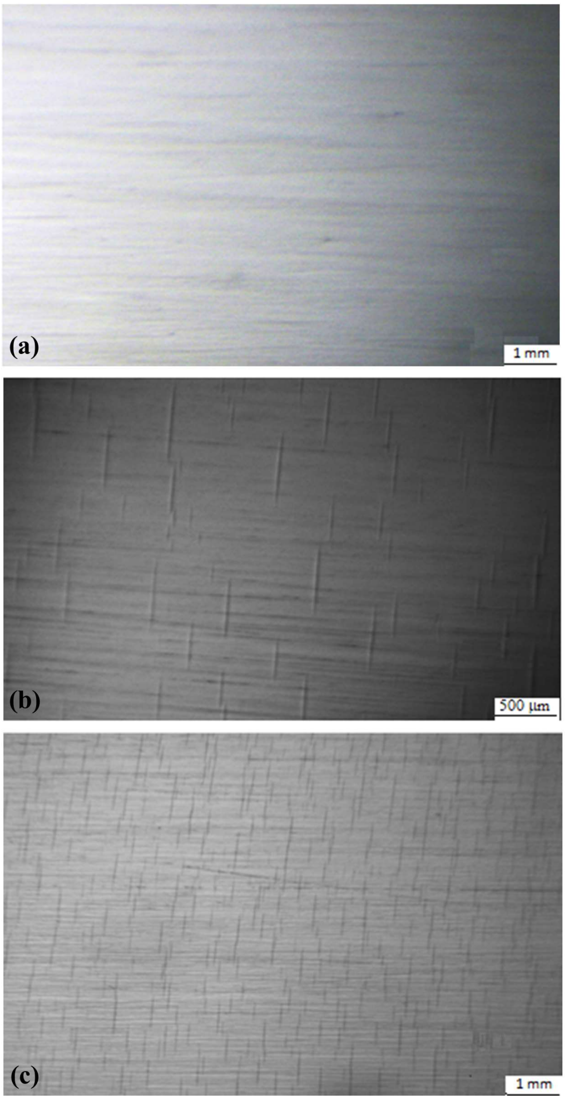

Fig. 7. Optical micrographs taken from the polished surface of pure PMMA under tensile test by 8.3×10^{-4} s^{-1} strain rate until indicated stresses at 25 ºC. (a) $\sigma = 0.25\sigma_y$; (b) $\sigma = 0.5\sigma_y$; (c) $\sigma = 0.75\sigma_y$.

and alignment, which can result in the formation of crazes. The extent and density of the crazes typically increase by increasing the stress values, as the polymer chains become more oriented and undergo greater strain. At very high stress values, the polymer material may ultimately fail catastrophically, with the crazes coalescing to form macroscopic cracks and fractures. The specific stress value at which this occurs depends on the polymer material and its properties, including its molecular weight, degree of crosslinking, and thermal properties.

Fig. 8 shows the stress-strain curve of pure PMMA/HA 5 at a constant strain rate. The position of the stop tensile test is labeled a, b, and c in the figure.

Fig. 9 shows optical micrographs taken from the polished surface of PMMA/HA 5 at different stresses. The pictures show the crazing mechanism is also visible for the composite sample. However, adding HA to PMMA causes the concentration of the crazing lines near the ceramic particles. As a matter of fact, the behavior of crazing in composite polymer materials is dependent on several factors, including the properties of the polymer matrix, the type and concentration of filler material, and the stress value applied to the material [25, 26]. The presence of filler material in the polymer matrix can have both positive and negative effects on the crazing behavior of the composite material. On the one hand, the filler material can increase the strength and stiffness of the composite, which can potentially reduce the likelihood of crazing at low stress values. On the other hand, the presence of filler material can also introduce stress concentrators or other defects into the material, which can increase the probability of crazing or failure under stress [24-26]. In the case of current research, HA is very rigid and has a high modulus of elasticity rather than PMMA, it may be more likely to introduce stress concentrations into the material and increase the likelihood of crazing. Similarly, if the filler material is very large or irregularly shaped, it may create steric hindrance effects that can increase the distance between polymer chains and reduce the intermolecular forces holding the material together, making it more susceptible to crazing. The results of current research indicate that increasing the percentage of HA content in the composite sample leads to a lower density of crazes on the lateral surface. When HA particles are absent, initial crazes appear in the matrix, due to stress concentration. By increasing stress, existing fibrils within the craze that

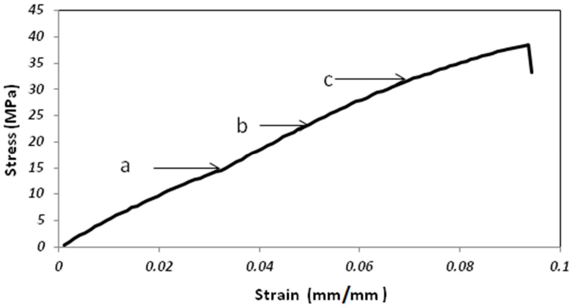

Fig. 8. Stress-strain curve of PMMA/HA 5 at the strain rate of 8.3×10^{-4} s^{-1}.

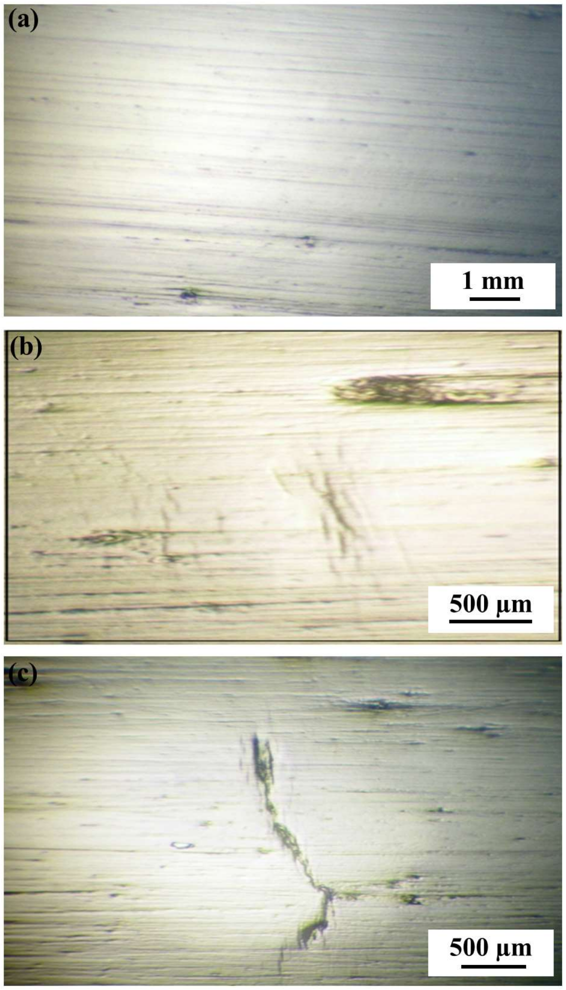

Fig. 9. Optical micrographs taken from the polished surface of PMMA/HA 5 under tensile test by 8.3×10^{-4} s^{-1} strain rate until indicated stresses at 25°C. (a) $\sigma = 0.25\sigma_y$; (b) $\sigma = 0.5\sigma_y$; (c) $\sigma = 0.75\sigma_y$.

act as the last links between the two edges of the craze transfer stress to its lateral side, allowing for a uniform distribution of crazes in the material. However, with the addition of HA particles to the matrix, poor connections with PMMA decrease the possibility of transmitting stress around the craze, causing the focus to be on the produced crazes. Therefore, increasing stress, instead of increasing the density of previous crazes, causes them to grow, break fibrils, and ultimately fracture the sample.

4. Conclusion

The Role of both the strain rate and the HA on tensile properties of the PMMA were investigated and the results are summarized as follows:

- Tensile strength of the PMMA depends strongly on both the HA and the strain rate, although the sensitivity of tensile strength to strain rate increases as the HA content increases. The results show that increasing the strain rate from 4.1×10^{-4} to 16.6×10^{-3} s^{-1} causes an increase in the tensile strength of PMMA to more than 26% while this promotion for PMMA/HA 10 is about 33%.
- The yield stress of the PMMA and its composites obey Eyring's model and activation energy for the yielding of PMMA decreases from 56.42 to 37.81 kJ/mol as HA content increases from 0 to 15 wt.%.
- The deformation mechanism of the PMMA and its composites are crazing and the intensity of the craze lines decreases as the HA content increases.

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Conflict of Interests

The authors declare no conflict of interest in this research.

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