

Study of Creep Property of Acrylic Resin Dental Material

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ABSTRACT

Creep response of denture base materials is being investigated in this study. The effect of both, temperature and hold stress variations, was taken into consideration. Temperatures of 25°C and 70°C were chosen to represent room temperature and high temperature similar to actual service temperatures. Different hold stresses were applied to investigate their effect on creep at the chosen temperatures. Hold stress values were determined to cover the region of yield point (slightly below, and above yield point). All creep tests were performed using tensile test specimens. The results showed that most specimens developed a typical creep curve quite similar to that developed by metals. Creep response was clearly affected by the increase of temperature. A clear increase in creep rate (shorter life) was associated with specimens tested at higher temperatures at the same hold stresses. In addition, increase of the hold stresses increased creep rate at the same temperature. Accelerated creep failure was due to polymer chains movement under hold load coupled with time. This movement became easier at higher temperatures, which contributed to a decrease in specimens lives with further increase in temperatures. It can be stated that increase of hold stress increases creep strain rates, while increasing temperature highly increases creep rate.

Key words: Acrylic resin material, creep, temperature

Introduction

An ideal denture base material is that possesses biocompatibility with the oral tissues, excellent esthetics, superior mechanical properties especially modulus of elasticity, impact strength, flexural strength, hardness, sufficient bond strength with artificial teeth and lining materials, rheological properties and dimensional accuracy (Meng and Latta 2005; Tuna *et al.*, 2008).

Creep is the deformation of solid material or viscous liquids under the influence of stress. In polymers, creep occurs by chains untangling and slipping relative to one another, because a polymer consists of long chain-like molecules in a tangled and coiled arrangement (Martin *et al.*, 2013).

Creep in plastics is one of viscoelastic properties, and information concerning the creep behavior of polymers is important as a guide to service performance. Polymers used in the construction of denture bases are subjected to masticatory loading during function. Thus it is important that there should be no change in dimensions during service (Stafford & Huggett, 1978). Long-term behavior, or creep, was a subject of research and study from many investigators.

Glantz and Bates, (1973) studied the creep behavior of four types of acrylic resins at protracted loading. They exposed the specimens to a stress of 12N/mm² for 30 hours under cantilever bending. The results showed that the heat cured resins have smaller initial deformation, less creep, quicker recovery, and less amount of permanent set than the dough and poured resins. Glantz and Stafford, (1973) concluded that repeated loading experiments using a stress level of 6N/mm² showed a similar pattern of time dependent deformation, but the permanent set increased with each successive loading cycle. Jagger and Huggett, (1975) concluded that the addition of cross linking agent in varying concentrations to a denture base material produced no increase in surface hardness, changes in creep and recovery were so small that they were of no clinical significance. Stafford *et al.*, (1975) found that the creep behavior of denture base resins is an important mechanical property that was not evaluated in standard dental material testing. Accordingly, they studied the development of a

compressive creep test based upon a standard hardness testing machine that showed a good correlation to three point creep behavior. They compared the result of this test with that obtained from transverse bend test. The stress applied in transverse bend test was equivalent to 76N/mm² corresponding to 125N/mm² at 23 °C for compressive test.

The authors concluded that all the tests showed that the polymers exhibited creep, and that the deformation on loading and the permanent set increased with each successive cycle. This behavior was similar in all tests described. Stafford and (Huggett, 1978) mentioned that the apparatus for creep testing was simple, and uniaxial tension has been a common technique experimentally.

Compressive creep, flexural creep, and shear creep techniques are also used. Two main requirements are necessary for accurate uniaxial tensile experiments. First, the force must be applied symmetrically along the geometric axis. And second, the extensometer must record the deformation in the specimen and yet, must not restrict it. The requirements for uniaxial compressive creep testing are similar. (Ruyter and Svendsen, 1980) concluded that the dough and pour type of auto polymerized materials have higher creep values than the heat polymerized materials. (Ruyter and Espeviks, 1980) studied the creep behavior of four types of denture base materials. The creep behavior was determined at 23°, 37° and 50°C in the compressive stress range of 5 to 75 MN/mm². The test lasted from a few minutes to 50 h depending on the creep rate with emphasis on secondary creep rate. They concluded that the creep rate was correlated to the processing conditions and the material composition. The lowest creep rates were found for the heat polymerized materials. Also two different creep mechanisms were evident, depending on the stress level. Below 20-40 MN/m² at 37°C, the creep was due to homogenous deformation. At higher stress levels, the creep was ascribed to heterogenous deformation mechanism. (Qysaed and Ruyter, 1989) studied the influence of type and quantity of five different cross linking agents on tensile creep properties of multiphase acrylic systems. The test was carried out at 37 and 50°C.

The investigation showed higher creep values for auto polymerized than for heat-polymerized materials. In heat-polymerized materials the creep curves showed little variation with type and quantity of cross linking agents at low stress levels. However, at higher stress levels the creep values decreased with increasing the quantity of cross-linking agents. The auto polymerized materials showed a more homogeneous structure and great variation in creep. Both, among the heat polymerized and auto-polymerized materials, the system with diethylene glycol-dimethacrylate clearly deviated from the others by showing higher creep values. (Sami *et al.*, 1990) studied the viscoelastic behavior of three types of polymer-based denture base material and eight volume fractions of aramide fibers reinforced acrylic resin using transverse bending test. The effect of the magnitude of stress on the creep behavior was also tested. They exposed the specimens to stress of 24N/mm² and 7 N/mm² on control and high volume percentage aramide reinforced polymer respectively, for 90 min under transverse bending. They concluded that the light-cured materials showed a similar behavior to the high impact material in the sense that its creep characteristics were higher than those of heat-cured acrylic resin, but they were lower than high impact materials. They also found that the aramide fibers reinforced acrylic materials showed a variable degree of improved creep behavior compared to the unreinforced heat cure acrylic with the 2.2 volume percent concentration showing higher values compared to the unreinforced acrylic. (William and Callister, 1991) found that uniaxial compression tests were more appropriate for brittle materials; as they provided a better measure of the intrinsic creep properties in as much as there is no stress amplification and crack propagation as with tensile loading. Creep tests on polymers are conducted in the same manner as for metals; that is, a stress (normally tensile) is applied instantaneously, which is maintained at a constant level while strain is measured as a function of time. (Sadiku and Biotidara, 1996) examined the creep behavior of acrylic denture base resins at room temperature and at different loading conditions (58.2g-558.2g). They concluded that the behavior of these resins was similar to the specimens of "Commerical perspex" (industrial heat cured resin) at room temperature over a period of 1 000 seconds. They also found that the "pseudo-elastic moduli" (Storage modules and time dependent young's modules) of the blends of polymethyl methacrylate/ polyvinyl chloride (PMMAlPVC) showed a significant increase compared with PMMA alone. The addition of the PVC powder to the heat cured resin increased the time-dependent elastic modulus.

This increase is advantageous in the production of denture base resins of improved mechanical properties. Craig, (1997) mentioned that when denture-base resins are placed under a load, they

deform (creep) with time. The lowest compressive creep rates are found for the heat polymerized materials. At low stress levels, the type and quantity of cross-linking agents have no major effect on creep. However at higher stress level, creep value decreases with increasing quantities of cross-linking agents. For heat-polymerized materials when the temperature is increased from 37 to 50°C, mode of failure changes from brittle to ductile.

Through the experimental course of this work the effect of temperature and/or applied load on creep were studied. This kind of study may help to determine the useful life service of acrylic resin materials subjected to load at higher temperatures which may be caused by creep under different loads.

Materials and methods:

Mold and specimen preparation:

The specimens, Fig. (1), were produced in molds made by investing aluminum pattern blocks of the required dimensions into gypsum. Heat-cured polymethylmethacrylate (Meliodent-Bayer Dental, Germany) was used for fabricating the test specimens. The proper monomer to polymer ratio as recommended by manufacturer was used (2.34 g powder to 1 ml liquid), and was thoroughly mixed and allowed to reach the dough stage in air-tight mixing Jars, then it was packed into the mold space. Two sheets of cellophane paper were used to cover the acrylic dough to prevent its adhesion to the lower mold surface during the trial closure. The cellophane papers were removed and the two halves were closed under pressure which was maintained until the specimens have been processed.

All specimens were subjected to the same curing cycle. The flask was submersed in tap water then the temperature of the water bath was raised to 72+ 1 °C and maintained for one hour followed by one hour boiling to ensure high degree of polymerization. The mold was then allowed to bench-cool to room temperature before deflasking. The specimens were stored in distilled water at room temperature for 48 hours.

Creep test

Specimens were divided into two groups corresponding to testing temperatures (28°C and 70°C). Eight specimens were tested at 28°C for holding stresses, 18.6 MPa, 23.31 MPa & 26.45 MPa. 24 specimens were tested at 70°C for holding stresses, 10. 75 MPa, 12. 71 MPa, 18.6 MPa, & 26.45 MPa.

Specimens shape and Dimensions

Specimens were dumb ell shaped (Fig. 1) with a parallel gauge portion of 30 mm (length), 5mm (width) and 3mm (thickness).

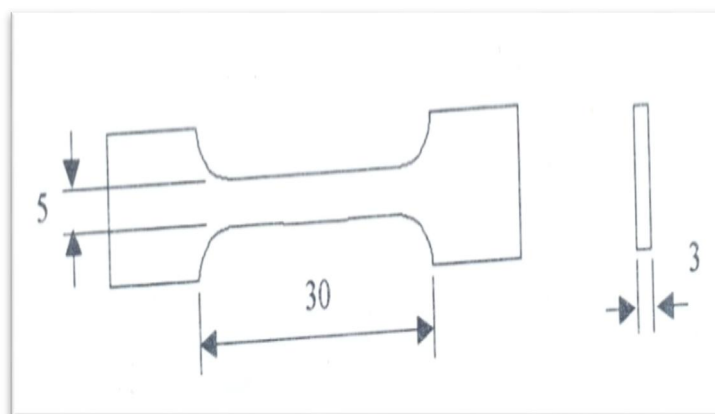


Fig. 1: Tensile specimen for creep tests, all dimensions in mm.

Creep apparatus

Constant tensile stress test (creep) was performed using SM 106 creep measurement apparatus illustrated-in (Fig.2). This apparatus is designed for demonstrating and investigating the creep characteristic at room temperature.

A well-prepared and calibrated heat coil was used to supply a uniform source of heat to keep the temperature constant within an error of $1\pm^{\circ}\text{C}$. This coil surrounds the specimen leaving almost no space for heat dissipation to ensure the uniformity of steady temperature during the time of load application of each individual test. The apparatus uses a simple lever to apply a steady load to the specimen.

The specimen was attached at one end to the lever mechanism by a steel pin and fixed at the other end to the fixed frame by another steel pin. Loads were applied to the lever arm by placing weights on the weight hanger, which is pinned to the; lever arm. The weight hanger has two pinning positions; the upper most is used to pin the hanger in the rest position while the lower hole is used to pin the hanger ill the loaded position. The lever arm has a mechanical advantage of 8. The mass of the arm is 0.4 Kg, the weight of the hanger is 0.16 Kg, and the pins used for pinning the weight hanger and specimens are 0.04 Kg each as illustrated in Fig. (2).

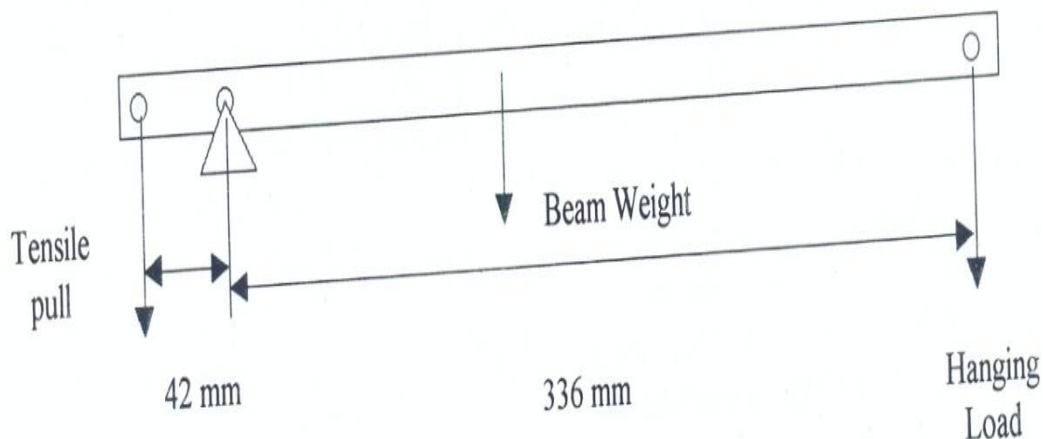


Fig. 2: Lever mechanism of creep apparatus

Measurements

The load on the specimens can be calculated by taking the moments about the pivot bearing. If the mass "m" is added to the weight hanger then the tensile pull "F" on the specimens was calculated using the following equation

$$F = (2.96 + 8m)g. \dots\dots\dots(1)$$

Where: Is acceleration due to gravity, m/see'

The specimen extension (strain) was measured by a dial test indicator (DTI) with an accuracy of 0.01 mm. The top of (DTI) is attached to the lever mechanism by means of a grooved plate, which is bolted to the lever arm. The arrangement is such that the groove in this plot is twice the distance from the pivot than that at the center of the specimen. Therefore the extension given by (DTI) is twice the actual extension of the specimen, which increases the accuracy of deformation measurements to 0.005mm.

Stresses applied

The yield strength of the material was determined through a conventional tensile test using universal testing machine. The curve, in Fig. (3) represents the averaged data of three tensile tests performed at similar conditions on the same tensile machine. The test speed was

kept constant during all tests at a rate equal to 5 mm1min. The yield strength was taken as the proof stress at 0.25% strain. This was established by drawing a parallel line to the initial straight portion of stress-strain curve starting from 0.25% strain till it intersected with the curve at a point that corresponds to the proof strength of the material which was taken to represent yield strength. Accordingly, stresses slightly below and above the yield strength were applied during creep testing.

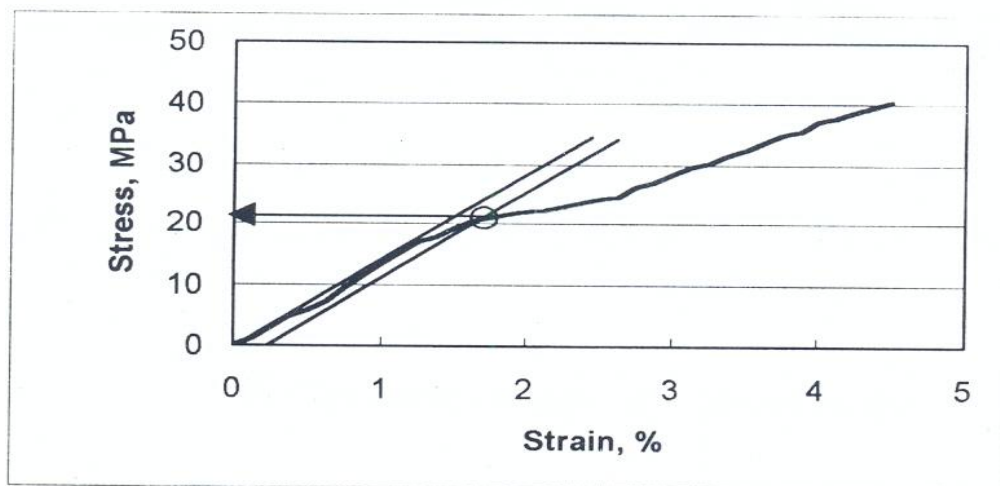


Fig. 3: Yield strength of PMMA represented on averaged tensile data as 0.25% stress

Results

The data from the creep experiments presented as creep strain versus time for the two chosen temperatures at different hold stresses are shown in table (1) and figs. (3-8). The creep curves obtained were similar to the typical creep curves that are obtained for metals. They display the three stages that can be distinguished; namely, primary, secondary and tertiary creep deformation schemes. When the polymer is stressed, an instantaneous elastic deformation occurs which is time independent. This instant deformation was subtracted from all the data of all tests to present creep strains only.

Table 1: Summary of creep testing results for PMMA

Hold stress, MPa	Temperature °C	Creep rate,1/min.	Total time before failure, min.
18.6	28	1×10^{-6}	11.800
23.3	28	3×10^{-6}	5.265
26.45	28	2×10^{-5}	245
10.75	70	2×10^{-4}	142.5
18.6	70	2.7×10^{-3}	8.85
26.45	70	9.5×10^{-3}	3.1

The first stage, known as primary creep, or viscoelastic response is time dependent. It represents a region of decreasing deformation rate, which depended on the hold stress and the test temperature. Testing temperature greatly affected the primary creep region as shown in Fig. (5) and Fig. (6). The primary region occupies a noticeable shorter range of the overall creep response with increasing the applied temperatures. The effect became more obvious with increasing the hold stress (comparing the response in Fig. (5) and Fig. (6) The primary creep rate was greatly decreased with the decrease of hold stress and emerged to a slower secondary creep as shown in Fig. (5).

The second stage of creep is known also as secondary creep or viscous response, a period of nearly constant creep rate. This region occupied most of the creep life of the whole curve of creep. It is usually referred to as steady-state creep since the rate of change of the creep curve curvature was

almost zero or was very small so it can be ignored. Figure (4) shows the complete creep curve of two tested specimens at 70°C. Even though testing duration of the two specimens was less than 10 minutes, both specimens creep data developed the complete typical creep curve. The secondary creep in each curve constituted more than 70% of the overall creep life.

For this reason, secondary creep constitutes the most attractive part of creep response to study. The slope of the second stage (secondary creep) of the creep curve (ds/dt) is usually referred to as the creep rate.

The third stage or tertiary creep followed the secondary creep stage for all tested specimens. However, the time after which tertiary creep and failure occurred was greatly affected by hold stresses and testing temperatures. Creep failure was developed in few minutes when a hold stress of 26.45 N was used at a temperature of 70°C, Fig. (4). Figures (7 and 8) show the effect of applied stress on the creep response at the two chosen temperatures. Room temperature creep (at 25°C) is shown in Fig. (7) at stresses of 18.6, 23.3, and 26.5 MPa.

It is apparent that creep responses were affected by the increase of hold stress levels. The higher the hold stress, the higher the creep rate (less time is required to produce same strain at higher hold stresses). A similar response was found also at higher temperature, Fig. (8). The increase of hold stresses contributed to an accelerated creep response at a temperature of 70°C. However, increase of testing temperature contributed to a great acceleration of creep strain. Using a hold stress of 12.7 MPa at 70°C produced a complete creep curve along a period of time of one day only. Increasing the hold stress to 26.3 MPa resulted in a complete creep curve along 3 minutes only.

A comparison of creep rates at different temperatures and different hold stresses are outlined in table 1. As shown in the table, the temperature increase has the most substantial effect on creep behavior. Increase of temperature from 28 to 70 decreased the lifetime of the specimens under creep with an order of approximately 100.

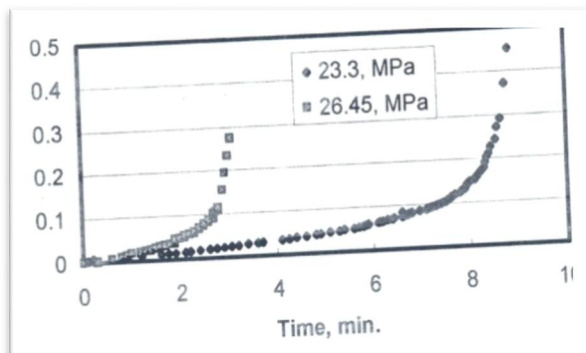


Fig. 4: Complete creep response at 70 C

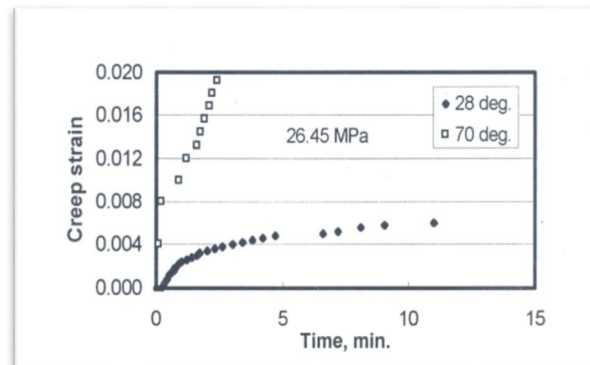


Fig. 5: The initial portion of creep curves at 26.5 MPa for two temperatures.

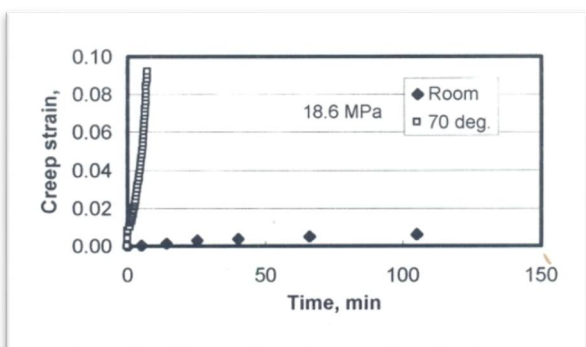


Fig. 6: The initial portion of creep curves at 18.6 MPa for two temperatures

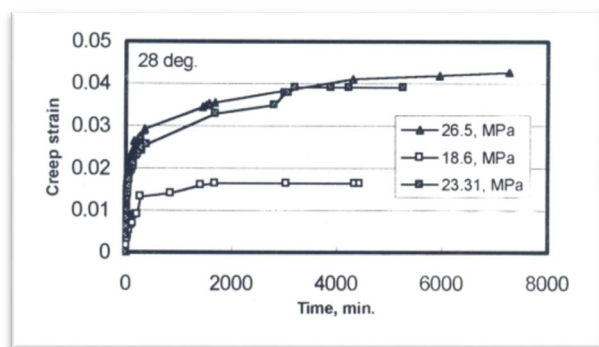


Fig.7: Creep curves at 28 C

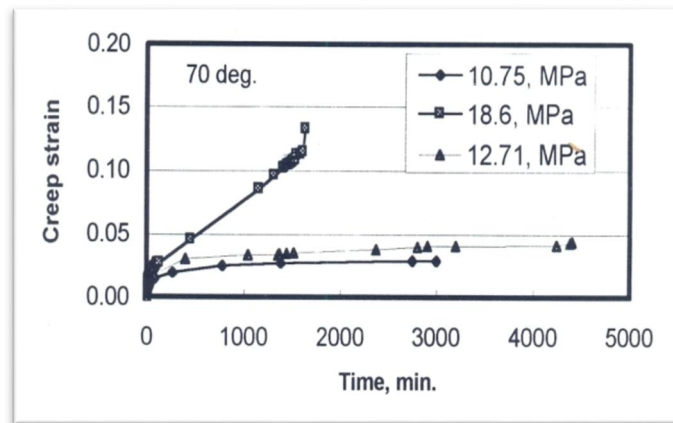


Fig. 8: Creep curves at 70 C

Discussion

Heat curing is the most popular method of processing denture base acrylics. The heat curing system was selected due to its adequate accuracy, good resistance to attack by solvents, and the laboratory cost is substantially less. Furthermore, the heat curing procedures resulting in dentures containing less significantly unreacted methyl methacrylate monomer (Hashem *et al.*, 2014 and Dalkiz *et al.*, 2012). Polymer deformation occurs progressively at a decreasing strain rate until it is constant or zero. Polymeric materials exhibit viscoelastic behavior; they do not exhibit purely elastic (ideal solids) or purely viscous (ideal liquid) behavior but a combination of both viscous and elastic properties in varying amounts. The rate of viscoelastic deformation depends on nature of the material, duration and temperature of exposure, and the magnitude of applied structural load. A typical creep test is performed by applying constant stress and monitoring the strain levels with time (Jia *et al.*, 2011).

The effect of hold stress increase on creep rate was much higher at lower temperatures compared to higher temperatures. The increase of hold stress from 18.6 MPa to 26.45 MPa at 28°C increased the creep deformation rate 20 times, while the same increase in hold stress contributed to an increase of 2.8 time only in creep rate at 70°C. It was expected that the increase in temperature would help increase the thermal motion of polymer chains in the polymeric system. This aspect is probably the basic reason for increased creep with increasing temperature. This is in agreement with Qysaed & Ruyter, 1989; Ruyter and Espevik, 1980). The contribution of hold stress of increased creep acceleration is in agreement with Ruyter and Espevik, (1980), who found that at highest stress level the material failed after relatively short loading times. This finding may be ascribed to an inhomogeneous deformation mechanism. This means that the strain is localized in certain local regions, i.e. the shear bands. In these shear bands strain softening may take place, resulting in increased creep rates. When in service, the dental components should never enter the tertiary stage of creep.

It is therefore the secondary creep stage, which is of prime importance as a design criterion. Components, which are subjected to creep, spend most of their lives in the secondary stage. So the material chosen for such components should have as small a secondary creep rate as possible. In general it is the secondary creep rate which determines the lifetime of a given component.

Conclusion

From this study, it could be concluded that:

- 1- Typical creep curves like those developed by metals were found for PMMA
- 2- Creep rate was greatly affected by temperature increase.
- 3- Creep rate increases with increasing hold stress.

- 4- The effect of hold stress increase was more remarked at lower temperatures than at higher temperature

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