

Creep Behaviors at 275 °C for Aluminum-Matrix Nano-composite under Different Stress Levels¹

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Abstract

Aluminum alloys, due to appropriate strength to weight ratio, are widely used in various industries, including automotive engines. This type of structures, due to high-temperature operations, are affected by the creep phenomenon; thus, the limited lifetime is expected for them. Therefore, in designing these types of parts, it is necessary to have sufficient information about the creep behavior and the material strength. One way to improve the properties is to add nanoparticles and fabricate a metal-based nano-composite. In the present research, failure mechanisms and creep properties of piston aluminum alloys were experimentally studied. In experiments, working conditions of combustion engine pistons were simulated. The material was composed of the aluminum matrix, which was reinforced by silicon oxide nanoparticles. The stir-casting method was used to produce the nano-composite by aluminum alloys and 1 wt.% of nanoparticles. The extraordinary model included the relationships between the stress and the temperature on the strain rate and the creep lifetime, as well as various theories such as the regression model. For this purpose, the creep test was performed on the standard sample at different stress levels and a specific temperature of 275 °C. By plotting strain-time and strain rate-time curves, it was found that the creep lifetime decreased by increasing stress levels from 75 MPa to 125 MPa. Moreover, by comparing the creep test results of nanoparticle-reinforced alloys and nanoparticle-free alloys, 40% fall was observed in the reinforced material lifetime under 75 MPa. An increase in the strain rate was also seen under the mentioned stress. It is noteworthy that under 125 MPa, the creep lifetime and the strain rate of the reinforced alloy increased and decreased, respectively, compared to the piston alloy. Finally, by analyzing output data by the Minitab software, the sensitivity of the results to input parameters was investigated.

Keywords: Creep, Aluminum alloy, Nano-composite, Nanoparticles, Regression model

1. Introduction

Aluminum-silicon alloys are widely utilized in various parts, especially in automotive and aerospace industries due to their proper properties. Besides, in these industries, some components are exposed to high temperatures and therefore, creep behaviors of utilized materials should be studied.

In the engine cylinder, the fuel energy is converted to thermomechanical forces. In fact, the amount of heat and pressure

increases dramatically in a short time. As a moving part of the combustion chamber, the piston is responsible for converting the released energy into the mechanical work. Therefore, the piston must have proper strength against mechanical and thermal loads. Based on thermomechanical loads and the actual piston operating temperature, the fatigue phenomenon (due to cyclic loading) and the creep phenomenon (due to high temperatures) can occur in the piston.



Aluminum alloys are known as one of the best materials for making piston alloys. Although aluminum is a soft and light metal, it is a strong material. Aluminum is a malleable metal as well as very durable and corrosion-resistant. Similarly, this non-magnetic, non-sparking element is the second malleable and the sixth antifouling metal. Aluminum alloys are metal alloys containing the pure aluminum and one or more elements to improve mechanical properties. High thermal conductivity makes the aluminum alloy a proper material to be utilized in the piston engine.

In this case, different researches have been performed on creep properties of aluminum-silicon alloys. In the following paragraph, a comprehensive review is represented for such a topic on creep testing of aluminum alloys.

Ishikawa et al. [1] studied the pure aluminum creep behavior at low temperatures. It was observed that the strain rate was related to the applied stress. In addition, a sensible relation was seen between cyclic stresses and the creep lifetime. Ishikawa and Kobayashi [2] worked on commercial aluminum-magnesium alloy creep behaviors. It was concluded that the minimum value of the strain rate was affected by the temperature and the applied stress tremendously. Dobes and Milicka [3] conducted a comprehensive comparison between the Al-Mg and Al-Zn alloy creep properties. They clarified that Al-Mg obstacle spacing in sub-boundaries was a value between the free dislocation distance and dislocation spacing. Requena and Degischer [4] investigated the unreinforced and reinforced AlSi12CuMgNi alloy isothermal creep resistance at 300 °C. The material was reinforced with different percentages of short alumina fibers. The embedding process of short fibers reduced the alloy creep rate by more than one order of magnitude. For 15% of reinforcements, higher creep resistance occurred based on higher defect density and also larger interface area. Li et al. [5] conducted the creep behavior prediction of 7075 and 2124 aluminum alloys at high temperatures by the continuum damage mechanic technique under the constant uniaxial tensile stress. This approach was simplified to describe for both first and second creep stages. Moreover, the third creep stage was analyzed by considering the stress in the model. An appropriate agreement was observed between experimental and modeling results. Fernandez-Gutierrez and Requena [6] investigated heat treatment effects on the creep resistance of the casting aluminum piston alloy. It should be noted that the aluminum piston alloy isothermal creep resistance, which was aged at 300 °C, was studied as a heat treatment solution function at 480 °C. It is noteworthy that using the air-cooling operation could increase the creep resistance. It was gently declined by increasing the solution time. Zhang et al. [7] represented a comprehensive model of tensile and creep behaviors of aluminum alloys and found the effect of the applied stress and the temperature on asymmetric mechanisms. Moreover, the creep model was validated mathematically for various asymmetric conditions. Wang et al. [8] systematically investigated the creep ageing behavior of the re-aged 7150 aluminum alloy. Their creep tests showed that the steady-state creep mechanism of the alloys was due to the dislocation climb, independent of boundary precipitates and the grain interior. However, a considerable increase in total creep deformation was observed during re-ageing. Moreover, by creep ageing at 140 °C for 16 hours, similar yield and tensile strengths and lower elongation were submitted. Ahn et al. [9] studied high-pressure die-cast AlSi10MnMg alloys and their creep behaviors at 100-300 °C, under the applied stress (64-217

MPa). They found that the micro-hardness was relatively low. These results were obtained due to the recovery and the recrystallization. Moreover, the minimum creep rate increased when higher stress was applied, which caused less failure time. Zhang et al. [10] studied the creep deformation of the rapidly solidified aluminum alloy. This research indicated similar or even better deformation performance compared to aluminum alloys. However, ageing at 525 °C for 200 hours caused the creep strength reduction. Golshan et al. [11] studied the influences of the heat treatment, the stress and the temperature on the creep behavior of AlSi12CuNiMg aluminum alloys. They observed higher creep lifetime and lower strain rate on heat-treated samples. In addition, a similar fracture behavior was observed by analyzing the fracture surfaces.

There were several published articles on the research about the material properties and also the creep behavior of metal-matrix composites. However, the number of these studies about the creep behavior of aluminum-matrix reinforced nano-composites was infrequent. The following articles represent details of reinforced composites by nanoparticles.

Pal et al. [12] investigated mechanical and tribological behaviors of aluminum alloys, reinforced by monometallic zirconium oxide nanoparticles. The positive effects of nanoparticles were revealed on both tribological and mechanical properties. An improvement was observed for the friction coefficient and also the wear resistance. Shuvho et al. [13] studied the material behavior of aluminum-matrix composites. They found higher hardness, tensile strength, and yield strength for the reinforcement by Al₂O₃, SiC, TiO₂ in 6063 aluminum alloy. It was also shown that adding more SiC particles led to an increase in the mechanical properties. Azadi and Aroo [14] investigated the aluminum alloy and the aluminum-matrix SiO2 nano-composite (2 wt.%) and their creep behaviors and failure mechanisms. Almost similar microstructure features were observed for both materials. Indeed, no considerable nanoparticles effect was seen on the base and nanoparticles-reinforced aluminum allovs microstructure. However, creep test results revealed that the lifetime decreased by nanoparticles. Since the stress concentration was observed due to nanoparticles, which led to more intermetallic phases in the microstructure and also flaked-shape silicon particles. Cadek et al. [15] analyzed creep data for aluminum alloys and aluminum-matrix composites with reinforcements. Significant effects of the reinforced material were observed for creep strengthening. However, no effective barriers were fabricated by particles against dislocation movements. Spigarelli and Paoletti [16] presented a model to illustrate the creep behavior of metals, which was validated in nano-sized particles reinforced composites. A relation was seen between the strain rate, the temperature and the stress. Moreover, they found reasons for lower creep rate of composites, compared to the base alloy. Gupta and Daniel [17] carried out the creep analysis for aluminum-matrix composites, using various weight percentages of Al3Ti particles. Their results illustrated higher activation energy and the stress exponent in composites, compared to the conventional alloy, according to the dislocation climb. Gong et al. [18] studied the creep behavior of the aluminummatrix composite at 250-350 °C and under 40-140 MPa. They reported the microstructure evaluation and the reason for higher creep activation energy in the composite. Zhao et al. [19] investigated mechanical properties of the Al-Mn-Mg alloy with



nano-TiC particles. These nanoparticles and also pre-straining effectively enhanced the material strength at low and high temperatures. It was claimed that small pre-stress could increase the yield strength and the concurrent precipitation suppressed the softening phenomenon due to the pinning process. This led to higher ductility and higher creep rupture lifetime. Bhoi et al. [20] investigated the aluminum-matrix composites reinforced by micro/nanoparticles. They observed better hardness and tensile strength value by using the nano-reinforcement in comparison to micro-reinforcing. Azadi et al. [21] investigated the impact of the loading rate on tensile features of the piston aluminum-silicon alloy, with and without silica nanoparticles. They discovered that increasing the loading rate could increase the yield stress and reduce the elongation in unreinforced samples. Bhowmik et al. [22] investigated the mechanical behavior of the aluminum-matrix composite reinforced by SiC/TiB₂. They revealed that the strength and the hardness of the reinforced material enhanced dramatically. Microstructure analyses indicated that SiC and TiB₂ contents were homogeneously dispersed throughout the aluminum matrix.

Based on the mentioned literature review, the novelty of this article is to investigate the creep behaviors of the aluminum-matrix nano-composite at 275 °C and under different stress levels. Therefore, creep testing was done and the fracture surface was analyzed.

2. Descriptions of the approach, the work methodology, and materials

In the present article, the effects of utilizing 1 wt.% of SiO_2 nanoparticles on the creep behavior and failure mechanisms of the aluminum-silicon alloy (AlSi12CuNi2Mg), which has been vastly used in engine pistons, was studied.

Standard creep samples were fabricated by the stir-casting method, which mold is shown in Figure 1. First of all, aluminum bars were melted at 750 °C. Then, nanoparticles with the amount of 1 wt.% SiO₂ (99% of the purity, with the dimension of 20-30 nanometers as the white amorphous powder) were added to aluminum melt. Notably, the planetary ball milling machine was used due to have a better distribution of nanoparticles in standard specimens.



Fig. 1. The casting mold for primary samples

Moreover, before casting, nanoparticles were wrapped in the aluminum foil. Then, the stir-casting process was done. The nanocomposite slurry was stirred for 2 min, at a constant rate of 100 rpm to achieve a homogenous distribution of nanoparticles in the matrix. In addition, all aluminum cylinders were quenched in the water at the temperature of 60 °C for 2 minutes, after casting. More details of the fabrication process could be seen in the literature [23]. Zolfaghari et al. [23] described about parameters of ball milling and also the casting process. Moreover, they proved the strengthening role of nanoparticles and showed their proper distribution in the aluminum matrix. Then, creep standard specimens were machined from casted cylinders, which is depicted in Figure 2.

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Fig. 2. Standard samples for creep testing

Creep tests were conducted by standard creep samples under the engine working conditions. Therefore, three creep tests have been conducted under different stresses and a constant temperature. The test temperature in this research was considered to be 275 °C. It should be noted that the temperature of the piston aluminum alloy is between 200 and 400 °C in the engine, besides the considered temperature of 300 °C in the literature [4,9,18]. The applied stress was also 75-125 MPa on 3 standard specimens. Creep testing conditions are mentioned in Table 2.

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The chemical composition of the studied piston aluminum alloy

Elements	Al	Si	Fe	Cu	Mg	Ni	Zn	Mn
Percent (%)	Base	12.50	0.41	2.4	0.74	2.2	0.07	0.3

Table 2

The creep testing conditions in this research

No.	Temperature (°C)	Force (Kg/10)	Stress (MPa)
1	275	9.6	75
2	275	16.0	125
3	275	19.2	150

All creep tests were conducted by SCT-30 creep testing machine, the Santam company. The machine capacity is 30 kN with the temperature of 1150 °C. The required forces were applied by weights, which were placed on the side lever manually. In addition, the heat chamber was located in the middle of the device. This device is shown in Figure 3. It should be noted that the ASTM-E139-11 standard was applied for creep testing.



Fig. 3. The SCT-30 creep testing machine

After testing, the fracture surface of failed samples was also analyzed by the field emission scanning electron microscopy to observe the failure mechanism. For a better and more accurate analysis of information, input parameters (the stress and the temperature) and output parameters (the strain, the strain rate and the lifetime) were analyzed both simply and logarithmically. The regression model represented input parameters and specimen features (nanoparticles) effects on output parameters by regression equations, written for the minimum strain rate, the final strain and the creep lifetime. Moreover, regression diagrams were obtained from the data analysis for input parameters and logarithmic output parameters. It is worth noting that nanoparticles effects are shown separately for each output parameter. In this research, a great agreement was observed between tests and modeling results.

The scanning electron microscopy (SEM) of the initial nanoparticles plus ball-milled aluminum powders could be seen in Figure 4. Moreover, the SEM image for the fabricated nanocomposite could be also observed in Figure 4, which showed a proper distribution of nano-particles in the aluminum matrix. To prove the existence of SiO₂ nano-particles in the matrix, the energy dispersive X-ray spectroscopy (EDS) was done and the result is presented in Figure 5. The EDS analysis demonstrated that on Point 1 (Figure 5), the maximum element was Si plus O, which indicated SiO₂ nano-particles, besides the diameter of 34-36 nm in the aluminum matrix.



Fig. 4. The SEM image for (a) initial SiO₂ nano-particle, ballmilled aluminum powders and (c) the nano-composite



Fig. 5. The SEM image for (a) the nano-composite and (b) the EDS analysis for Si and O elements

3. Descriptions of achieved results

3.1. Creep Testing Data

Creep tests results include the creep lifetime (minutes), the minimum strain rate (per minutes) and the strain at different equivalent stresses are shown for the aluminum alloy with nanoparticles in Table 3. According to the obtained results, it is clear that by enhancing the applied stress from 75 to 125 MPa, in aluminum alloys with nanoparticles, the creep lifetime decreased from 902 minutes to 57 minutes, which was 94% less than the unreinforced aluminum alloy. Moreover, by re-increasing the stress from 125 to 150 MPa at the same temperature conditions, almost 99% decline was observed and the creep lifetime reached less than 1 minute. Such an effectiveness of the stress on the creep behavior of aluminum alloys was also presented in the literature [1-2]. As the stress increased, the aluminum alloy minimum strain rate raised; therefore, a downward trend for the creep lifetime was observed. The results presented in the literature [14], on the aluminum-piston alloy with and without nanoparticles, illustrated that the minimum value of the strain rate enhanced, when the

applied stress level enhanced, which was similar to the results, obtained in this research. Moreover, strain-time and strain rate-time curves are presented in Figure 6. It should be noted that the first region of creep data was not observed in figures. The same pattern was also observed in creep results in the literature [14]. As a note, they conducted creep tests for the aluminum alloy without nanoparticles under a constant temperature (275 °C) and different stresses. According to the results, lower creep lifetime and higher strain rate were observed for the reinforced aluminum alloy with nanoparticles under 75 MPa of the applied stress. The creep lifetime decreased from 1517 minutes (based on data in the literature [14]) to 902 minutes by nanoparticles. Moreover, the minimum strain rate in the sample with nanoparticles was 63%, higher than the minimum strain rate in the unreinforced sample. However, under loading conditions with the same temperature and the stress of 125 MPa, longer lifetime and higher minimum strain rate were observed for specimens with nanoparticles.

Table 3				
The creep	test results	for the	nano-comj	posite

Number	Temperature (°C)	Stress (MPa)	Creep lifetime (min)	Minimum value of strain rate (1/min)
1	275	75	901.77	0.000038
2	275	125	57.29	0.000613
3	275	150	0.33	0.009831



Fig. 6. Curves of (a) strain-time and (b) strain rate-time for creep testing at 275 °C



Such an improvement in material properties was also reported by Pal et al. [12]. They strengthened the wear behavior of aluminum alloys, reinforced by monometallic zirconium oxide nanoparticles. Shuvho et al. [13] improved mechanical properties of aluminum-matrix composites by adding SiC particles.

According to the literature [14], the sample lifetime without nanoparticles was reported 14 minutes in the same conditions. Simultaneously, under the same stress level, the creep lifetime increased by 307% to 57 minutes when nanoparticles were added. Besides, a lower minimum strain rate was observed when nanoparticles were added to samples.

3.2. Regression Model

The regression model could be helpful to analyze data more accurately. The regression model represents the influence of each input parameter on the creep lifetime, the final strain, and the strain rate. The P-Value and R^2 values for the regression model indicate the accuracy of curve-fitting. Moreover, the P-Value of each input shows its effectiveness on the output parameters. The P-Value and the R^2 value should be less than 0.01 and more than 90%, respectively, to consider the input parameters as effective ones.

The best results were submitted by using the standard form of input parameters and logarithmic values of output parameters. For the final strain, the P-Value was 0.004 and the R² value was 95.25%, and the effective parameter was the applied stress. For the minimum strain rate, the P-Value was 0.011, the R² value was 92.38%, and the effective parameter was the nanoparticles effect. In addition, for the creep lifetime, P-Value and R² values were 0.006 and 94.32%, respectively and no effective parameter was found for this output factor. Figure 7 represents the sensitivity analysis, using the Minitab software. It shows the effect of the applied stress level, the temperature and nanoparticles on the creep lifetime, the final strain and the strain rate. As noted, higher strain rate and lower creep lifetime were expected when more stresses were applied. In addition, by increasing the temperature, higher strain rate, a constant strain and lower creep lifetime were expected. Longer creep lifetime and the strain rate and a slight decline in the strain were anticipated. Table 4 demonstrates a comparison between experimental and modeling results. An appropriate agreement was observed between experimental and modeling results.

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A	co	mparison	between	regression	modeling	and	exp	erime	ntal	data
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Reference	variations	Liletime	Strain	Strain rate	
Experimental	Increase in	Decline	Unknown	Increase	
-	stress level		benavior		
	т.		Decline-		
Modeling	Increase in	Decline	increase-	Increase	
	stress level		decline		

The application of using the Minitab software was previously proven by Balachandran et al. [24], Kumar et al. [25] and Sreedev et al. [26]. Balachandran et al. [24] investigated NBR composites and tried to optimize composite properties by the regression analysis. For a similar aluminum-matrix composite, Kimat et al. [25] studied the effect of B₄C/Al₂O₃ on the wear behavior of Al6.6Si-0.4Mg alloy. They found the second-order polynomial equation and the response surface by the regression analysis in Minitab software. Sreedev et al. [26] determined the significance of cobalt addition on the wear characteristics of the aluminum alloy by analyzing with a response surface methodology.

As it could be seen in Figure 7 (a), the nanoparticles effect was more highlighted for the minimum strain rate, compared to the final strain and the creep lifetime. Cadek et al. [15] showed a significant influence of the reinforced material on aluminum alloys for creep strengthening. Shankar et al. [27] found the influence of the T6 heat treatment on the tribological behavior of cast Al-12.2Si-0.3Mg-0.2Sr alloy by the response surface methodology. In another similar work, Anilkumar et al. [28] checked the heat treatment impact on the wear behavior of Al-14.2Si-0.3Mg-TiC composite, again using the response surface methodology. Their results depicted that by increasing the ageing temperature, the ageing time and the load, a variation was observed in wear properties of the material.





3.3. Fracture Surface

Figure 8 represents the field emission scanning electron microscopy (FESEM) images, which showed the crept sample fracture surface. Based on these FESEM images, the fracture



behavior of the nano-composite was brittle. In addition, both quasicleavage and cleavage patterns were visible on the fracture surface. Moreover, pores and micro-cracks were also observed on all specimen fracture surfaces. The same microstructure was observed by Jiang et al. [29]. They conducted tensile creep tests on the 8030aluminum alloy under the stress of 50, 70 and 90 at 150 °C. Based on the fracture surface, the grain size reduced and dislocations raised when higher stresses were applied. Moreover, increasing the level of the stress could accelerate the formation of dislocations. Tangled dislocations caused walls to form grains that turn into smaller grains. To investigate the nanoparticles effect on the material microstructure, creep failure surfaces of the piston aluminum alloy specimens were analyzed and compared to the piston aluminum alloy samples with nanoparticles.



Fig. 8. The fracture surface of creep test at 275 °C and under (a) 75 MPa, (b) 125 MPa, and 150 MPa of the applied stress (Features included cleavage, guasi-cleavage, tear-ridge marks, micro-cracks and casting properties)

Cadek et al. [15] indicated that the particle reinforcement had no effective barriers against dislocation movements. Gupta and Daniel [17] demonstrated higher activation energy for the creep behavior of aluminum-matrix composites, reinforced by Al₃Ti particles, which was due to the dislocation climb in the microstructure of the material. Azadi et al. [30] observed the same phases for the piston aluminum alloy, reinforced by SiO₂ nanoparticles. Smaller grain size was observed for the reinforced material than the base alloy, which demonstrated an acceptable agreement with this article results. Khisheh et al. [31] and Rashnoo et al. [32] reported the distribution maps on the fracture surface of aluminum-matrix nano-composites, with the energy dispersive X-ray spectroscopy (EDS). They found that the first failure mechanism was as a reason of the Si phase in the matrix. The second failure mechanism was the existence of the intermetallic phase. Moreover, micro-cracks could be also seen on the sample fracture surface around such an inhomogeneous intermetallic phase.

Again, the same microstructure was observed by Azadi and Aroo [14] in the AlSi12CuNiMg piston alloy. The statistical analysis for Si particles indicated that the silicon phase size for the materials, the nano-composite and also the aluminum alloy, was calculated to be about 1180 and 3154 µm², respectively. Therefore, it could be claimed that by adding nanoparticles to the material, the second phase size of the aluminum matrix was reduced. High porosities were also seen on the fracture surface of the first sample with 75 MPa of the applied stress. This could provide a reasonable explanation for the shorter creep lifetime of the sample with nanoparticles. The FESEM image of the related sample is depicted in Figure 9. Azadi et al. [21] studied the fracture surface of reinforced aluminum alloys. They revealed that by enhancing the loading rate in tensile testing and adding silica nanoparticles, the material brittleness increased. A great agreement could be followed by comparing Figures 8 (b) and (c), which indicated smaller grains under higher stress load.

Consequently, the occurrence of the porosity in the analyzed samples indicated that the nano-composite was not prepared properly. In order to complete such a research, it is suggested that the casting process could be done again to have high-quality samples and then checking the creep behavior of the nanocomposite.



Fig. 9. The FESEM image of the sample with nanoparticles under 75 MPa including porosities (yellow marks on the image indicated the casting porosity)

4. Conclusions

In this article, tensile creep tests were conducted for 3 samples, which were reinforced with nanoparticles, at a constant temperature (at 275 $^{\circ}$ C) and under applied stresses from 75 to 150 MPa. The creep lifetime, the final strain and the strain rate of samples have been

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studied as output parameters. The creep behavior parameters were compared with unreinforced samples.

Experimental data could be described and listed as follows,

- Lower creep lifetime was indicated when the stress lever increased from 75 MPa to 125 MPa. According to strain-lifetime and strain rate-lifetime charts the creep lifetime decreased over 94 % by increasing the stress level.
- Nanoparticles effects were seen by strain-creep lifetime and strain rate-lifetime charts. The creep lifetime decreased when nanoparticles were added to the specimen under 75 MPa. However, the creep lifetime under 125 MPa raised in comparison to specimens without nanoparticles.
- Primary creep stages were missed in the nano-composite based on the refinement of grains, which occurred during casting. In this regard, the main reason could be the addition of nanoparticles.
- Based on the analysis, it was found that the amount of the strain in the aluminum alloy was related to the stress applied during testing. Moreover, no sensitivity was seen for the temperature and nanoparticles. The strain rate of the aluminum alloy showed a sensitivity to the stress and nanoparticles.

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