Influence of Cryogenic Temperature (CT) on Tensile Properties and Fatigue Behavior of 2024-Al2O3 Nanocomposites

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Abstract. The stir casting process used to manufacture the AL/ Al₂O₃, AA2024 with matrix material alumina Al₂O₃ at different weight percentage ratio 0.3, 0.5, 0.7 and 0.9%. Were fabricated by using cryogenic temperature (CT). It was observed that the maximum enhancement is occurred at nanocomposite containing 0.9% Al₂O₃ which shows improvement percentage of 9.9%, 14.63% and 14.28% for ultimate strength, yield stress and Brinell hardness respectively and the ductility is reduced by 34.57% compared to 0% by weight AA2024. It clearly shows that all the mechanical properties are improved by increasing weight percentage of Al₂O₃ but the best properties are obtained at 0.9% Al₂O₃ nanocomposite. The fatigue S-N curve tests were done for all the specimens having average of roughness between 0.2 to 0.5Mm. The fatigue endurance limit of the 0.9% wt. Al₂O₃ composite into two cases without (CT) and with (CT) is taken at 10⁷ cycles. The fatigue limit at 10⁷ cycles increases from 83 MPa for without (CT) to 89.6 MPa with (CT) resulted in 7.95% fatigue limit improvement.

1. Introduction

Among the metal matrix composites particle-reinforced aluminum matrix composites have attracted substantial interest among researchers as engineering materials due to their mechanical properties like strength to weight ratio and improved hardness, superior to those of general aluminum alloys. Some of the typical applications of these MMCs are bearings, automobile pistons, cylinder liners, piston rings, connecting rods, sliding electrical contacts, turbo charger impellers, space structures, etc. [1]. Different methods are used to fabricate MMCs one of them is stir casting method, it is the most commonly used due to have advantages such as the ability to mass production, lower processing cost, simplicity in operation, easier control over matrix composite, improve particle matrix bounding [2].

Al-alkawi et al. [3] used MMCs of 7075 AA with different wt% of Al₂O₃ at average size of 10nm, were manufactured using stir casting method. The results showed that when adding particles of Al₂O₃ the mechanical properties of nanomaterial were improved such as Brinell hardness, yield stress (σy), ultimate strength (σu) and ductility. Maximum improvement in hardness BHN, yield stress and ultimate strength were observed by weight percentage 0.2 Al₂O₃, While the lowest value is obtained of ε % at weight percentage 0.2 Al₂O₃.

Kumar K. et al. [4] used Electromagnetic stir casting process and mechanical stir casting combined with a process of Hybrid Casting. AA 2024 reinforced with 1 % wt. Al₂O₃ nanocomposite was manufactured. The composite was tested by microscopy (SEM) and tensile testing. The results of tensile strength has been improved by 43% from Al2024 with 1 % wt. Al₂O₃ nanocomposite when compare it with the Al2024 alloy.

Ashwath p. et al, [5] studied the manufacture of aluminum alloy AA-SiC and AA- Al₂O₃ with weight percentage (3, 6 and 9) of SiC and Al₂O₃ with 10 μm average particle size. Aluminum alloy 2024 reinforced with 6% wt. of Al₂O₃ samples were showed improved hardness results, stress-strain...
and strength behaviour. Aluminum alloy 2900 reinforced with 6 wt% Al₂O₃ showed good formability and ductility properties closer to 2024AA. The results showed that frictional coefficient behaviour of AA 2900 with 6 wt% Al₂O₃ was better than of AA 2024.

S.A. Sajjadi et al. [6] fabricated Micro composites and nanoparticles (A356/ Al₂O₃) with a different percentage of particles wt. using two melting techniques such as compo-molding and stir- molding. Pressure tests, stiffness, hardness and tensile strength were conducted to determine the mechanical properties of compounds. The results of the study showed that mechanical properties (ultimate tensile, yield and compression strength) are improved when added alumina (nano, micro).

Gao S. et al [7] studied deep cryogenic treatment used to AA5A06 at temperature (-155°C) for (4H,8H,10H)) respectively. The results showed that after the use of cryogenic treatment (CT), the mechanical properties of 5A06 were significantly improved. The main object of this work is to study the mechanical properties of Al₂O₃ particle-reinforced 2024 aluminium alloy composites and investigate the effect of cryogenic temperature on mechanical properties of the same material.

2. Experimental Work

Metal matrix composites materials (MMCs) are two or more materials mixed with different properties, one material is called matrix - phase , the other one is called reinforcement material, in this study AA 2024 with chemical composition at table (1), and reinforcement material aluminium oxide Al₂O₃ with chemical composition at table (2) are used.

| Table 1. Chemical Composition of AA 2024 |
|------------------------------------------|
| Component | % Si | % Fe | % Cu | %Mn | % Mg | %Cr | % Zn | %Ti | % other | % Al |
| Standard [8] Max. | Max. | 3.8- | 0.3- | 1.2- | 0.1 | 0.15 | 0.15 | Max. |
| | 0.5 | 4.9 | 0.9 | 1.8 | | | | 0.15 |
| Measured | 0.48 | 0.46 | 4.2 | 0.52 | 1.48 | 0.8 | 0.11 | 0.11 | - |

| Table 2. Chemical Composition of Al₂O₃ |
|----------------------------------------|
| Chemical Composition | α-Alumina | Fe₂O₃ | TiO₂ | CaO | Other magnetic material |
| Reference [9] (wt.%) | ±93 | ±0.8 | ±1.8 | ±1.1 | ±0.2 |

2.1 Mixing

The stir casting process used to manufacture the AL/Al₂O₃. AA2024 with matrix material alumina Al₂O₃ at different weight percentage ratio 0.3, 0.5, 0.7 and 0.9%. Were fabricated by using stir casting method. This method involves several steps that can be summarized as follows [2]:

1. Preparing the specimens by cutting into cubes (1-2) cm3 and washed it with alcohol, then washed with steam of hot air at a temperature 100°C after that dry parts.
2. Heated parts by an electric heater at 200°C.
3. Lift the oven covers and carry the resulting parts of the heating process, then close the oven cover tightly. Then, withdrawn air from the oven using vacuum.
4. After pumping the furnace with Argon gas, it is heated to 800°C
5. The alumina (Al₂O₃) are heated to 200°C.
6. The nanoparticles are added to the molten AA2024 through the gas pump.
7. Raised the temperature of furnace is over melting temperature of Al. about 850°C then cooled it less 650°C.
8. Four minutes are designed for stirring and with stirring speed 450 rpm.
9. Mixing is heated to 850±10°C
10. Finally, pour the molten into appropriate molds to obtain an aluminum rod.

![Figure 1. Block diagram of stir casting process](image_url)

**2.2 Cryogenic Treatment**

Deep cryogenic treatment (DCP) is a processing that can be used to improve the mechanical properties of components, this process have several advantages to improve fatigue resistance, hardness and grain size. The block diagram of DCP represents in figure (2). DCP was applied to samples of 2024Al/Al₂O₃ nanocomposite that fabricated at several step:
1. Wash the samples several times with methanol then dried the samples
2. Used cooling instrument type SKL500u
3. The sample put in vertical position in holder of the cooling chamber
4. The samples was slowly cooled to avoid thermal shock by using to cooling chamber
5. The cooling process till the samples reach at (-160°C) had been taken at 4 hour.
6. Hold the samples at (-160°C) for 24 hour
7. The samples reheated slowly without thermal shock to ambient temperature
8. The sequence of this treatment was carried out in vacuumed environment.

![Figure 2. Schematic diagram of deep cryogenic treatment processing](image_url)

[10]
2.3 Sample dimensions and test apparatus

Figure (3) shows the fatigue specimen According to America Standard Specification (ASTM) which means the (American Society Testing and Materials).

![Figure 3. The Fatigue Specimen According to ASTM](image)

A rotational-type fatigue test device was used (Scheck pun rotary bending machine) as shown as in figure (4) and with a speed of 1420 rpm so as to test the matrix material alumina Al$_2$O$_3$ specimens. 24 specimens were tested for each level of stress and extraction to eliminate the scattering that occurs in fatigue test, this is the usual context in fatigue tests.

![Figure 4. The Fatigue Test Device Type (Scheck pun rotary bending machine)](image)

3. Results and discussion

3.1 Mechanical Properties

Table (3) and figure (6) gives the tensile strength and mechanical properties results with different weight percentage of the reinforced material under to cases of testing (RT) and (CT).

| Material  | Without (CT) | With (CT) |
|-----------|--------------|-----------|
|           | *UTS (MPa)  | **YS (MPa) | BH | E(GPa) | $\varepsilon$ | *UTS (MPa) | **YS (MPa) | BH | E(GPa) | $\varepsilon$ |
| AA2024    | 182          | 82         | 58 | 68.4   | 11        | 186          | 87         | 63 | 69     | 10.7 |
| 0.3% Al$_2$O$_3$ | 188       | 86         | 60 | 69     | 10        | 201          | 90.5       | 65 | 70     | 8.4  |
| 0.5% Al$_2$O$_3$ | 192       | 89         | 62 | 69.7   | 9.2       | 207          | 91         | 68 | 70.3   | 8    |
| 0.7% Al$_2$O$_3$ | 196       | 91         | 64 | 70     | 8.6       | 211          | 95         | 70 | 70.6   | 7.3  |
| 0.9% Al$_2$O$_3$ | 200       | 94         | 66 | 70.2   | 8.1       | 215          | 98         | 72 | 71     | 7    |

* (UTS) Ultimate Tensile Strength
** (YS) Yield Stress

Mechanical properties can be determined by performing a tensile test during which a homogenous standardized test sample is subjected to a controlled the applied load (P) and deformation (\(\Delta L\)).
Considering the initial dimensions of test sample, cross-section area ($A_o$) and initial length ($L_0$) then the average linear stress ($\sigma$) can be calculated by:

$$\sigma = \frac{P}{A_o}$$  \hspace{1cm} (1)

And strain $\varepsilon = \frac{L_f - L_0}{L_0} = \frac{\Delta L}{L_0}$ \hspace{1cm} (2)

Where $L_f$ is the final length of failure

The stress ($\sigma$) and strain ($\varepsilon$) can be plotted to outline the engineering stress-strain curve. Fig.(5) shows the stress-strain experimental curve of 0.9wt.% Al$_2$O$_3$ in both (RT) and (CT).

**Figure 5.** Engineering Stress-Stain curve for 0.9 wt.% Al$_2$O$_3$ composite at (RT) and (CT)

From the results presented in table (3), it is seen that there is a significant improvement in mechanical properties and hardness after (CT) treatment and the maximum enhancement is occurred at nanocomposite containing 0.9% Al$_2$O$_3$ which shows improvement percentage of 9.9% and 14.63% for UTS and YS respectively and the ductility is reduced by 34.57% compared to 0% by weight AA2024. It clearly shows that all the mechanical properties are improved by increasing weight percentage of Al$_2$O$_3$ but the best properties are obtained at 0.9% Al$_2$O$_3$ nanocomposite show fig(6). These improvement may be coming from reinforced particles are uniformly distributed on AA2024 metal matrix and this parameter is a great drawback in producing metal matrix composite [11]. The thermal mismatch between matrix and Al$_2$O$_3$ is the main factor for increasing the density of dislocation leading to increase the composite mechanical properties [12].

**Figure 6.** Mechanical Properties (UTS and YS) of 2024 Al. alloy with different percentage of Al$_2$O$_3$ at (RT) and (CT).
Modulus of elasticity (E) defined as (Stress/ Strain) and it is measured of the stiffness of material (E = \frac{A_0}{L_0}) and it is used for calculating structural deflections. Ref [13] mentioned that the modulus of elasticity is slightly high at low temperature compared to ambient temperature and it drops at elevated temperature.

It is observably determined that (E) increases when the temperature decreases. Generally (E) under (CT) is greater than that of modulus of elasticity at ambient temperature. For composite containing 9 wt.% Al₂O₃ the value of (E) increased from 70.2 to 71GPa without and with (CT) respectively showing 1.139% improvement.

The variation of (E) verses the amount of Al₂O₃ under ambient temperature and (CT) can be illustrated in fig. (7).

![Figure 7. Young Modulus (E) against wt.% of Al₂O₃ for room temperature (RT) and (CT).](image)

The ductility of aluminum alloys depends on temperature and their crystal structure. Aluminum and its’ alloys are ductile even at low temperatures. This property provides using aluminum alloys in cryogenics applications. The experimental results revealed that the ductility of matrix and composites reduced at (CT) compared to (RT) as shown in fig. (8).

![Figure 8. Ductility VS wt. % of Al₂O₃ at (RT) and (CT).](image)

It is clear that the behaviour of AA2024 changes greatly when tested at (CT) compared to (RT) behaviour. This finding is greed well with Ref [14].
3.2 Fatigue S-N Curve Results

There are many factors that influence the S-N curve performance which are (temperature, surface treatments, loading ratio, cycling frequency and type of reinforcement) [15]. The fatigue S-N curve tests were done for all the specimens having average of roughness between 0.2 to 0.5Mm. four applied stresses were selected depend on the mechanical properties and for each stress level there are three specimens were tested with stress ratio (R= -1),12 specimens were examined without (CT) and 12 specimens with (CT) and the result have been listed in table (4) and graphically displayed in form of constant S-N curves as shown in fig.(9). The power law formulas which describe the fatigue behaviour have good correlation factor ($R^2$) as given in table (4) which indicated that the experimental results are well explained by power law equation. The fatigue endurance limit of the 0.9% wt. Al$_2$O$_3$ composite into two cases without (CT) and with (CT) is taken at $10^7$ cycles. The fatigue limit at $10^7$ cycles increases from 83 MPa for without (CT) to 89.6 MPa with (CT) resulted in 7.95% fatigue limit improvement.

Table 4. Fatigue S-N curves Results of AA2024

| Spe. No. | Nano wt% | Applied stress (MPa) | Nf (cycles) | Spe. No. | Applied stress (MPa) | Nf (cycles) |
|----------|----------|----------------------|-------------|----------|----------------------|-------------|
| 1        | 0.9%     | 120                  | 980         | 13       | 140                  | 2600        |
| 2        | 100      | 140                  | 1020        | 14       | 140                  | 3100        |
| 3        | 100      | 140                  | 1200        | 15       | 140                  | 1800        |
| 4        | 120      | 120                  | 35600       | 16       | 120                  | 82800       |
| 5        | 100      | 41800                | 38000       | 17       | 120                  | 90000       |
| 6        | 100      | 1008000              | 1256000     | 18       | 120                  | 78800       |
| 7        | 100      | 1120000              | 1256000     | 19       | 120                  | 1460000     |
| 8        | 120      | 1008000              | 1120000     | 20       | 100                  | 1825000     |
| 9        | 90       | 1256000              | 1256000     | 21       | 100                  | 2120000     |
| 10       | 90       | 3680000              | 1008000     | 22       | 90                   | 5680000     |
| 11       | 4860000  | 3680000              | 1120000     | 23       | 90                   | 4860000     |
| 12       | 3380000  | 3380000              | 1256000     | 24       | 90                   | 3980000     |

$\sigma_f = 205N_{fav}^{-0.056}$

$R^2 = 0.9865$

$\sigma_f = 221N_{fav}^{-0.056}$

$R^2 = 0.9764$

Fatigue endurance limit (MPa)

83

89.6
4. Conclusion
1- The effect of (CT) treatment on mechanical properties, hardness and fatigue behavior of AA 2024 matrix and nanocomposites has been examined through experimental investigation.
2- The stir casting technique was successfully adopted in the fabrication of AA 2024 -Al₂O₃ nanocomposites.
3- Mechanical properties and hardness of the AA2024-Al₂O₃ nanocomposites increased as compared to that of matrix AA2024 at room temperature.
4- The (CT) treatment show an increase in mechanical properties and hardness but the ductility of matrix and composites decrease significantly with increase the amount of Al₂O₃. The improvement in (UTS), (YS) and hardness due to (CT) treatment were increased by 9.9% ,14.63% and 14.28%, respectively while percentage elongation decrease with (CT) treatment.
5- It has been demonstrated that the (CT) treatment is an effective technique to improve the fatigue constant S-N curve of 0.9% Al₂O₃ composite.

5. References
[1] Purohit, R., Rana, R.S. and Verma, C.S., 2012. Fabrication of Al-SiCp composites through powder metallurgy process and testing of properties. International Journal of Engineering Research and Applications, 2(3), pp.420-437.
[2] Hussain J.M. Al-Alkawi , Maha N. Abdulridah and Raad H. Majid,2019. Fatigue Strength of Nanocomposite Under High Temperature. Journal of Engineering and Applied Sciences, 14 (14), PP 4742-4746.
[3] Al-Alkawi, H.J.M., Al-Rasiaq, A.A. and Al-Jaafari, M.A.A., 2017. Mechanical Properties of 7075 Aluminum Alloy Matrix/Al2O3 Particles Reinforced Composites. Engineering and Technology Journal, 35(3 Part (A) Engineering), pp.239-245.
[4] Kumar, K., Verma, D. and Kumar, S., 2014, December. Processing and tensile testing of 2024 Al matrix composite reinforced with Al₂O₃ nano-particles. In 5th International & 26th All India Manufacturing Technology, Design and Research Conference (AIMTDR 2014).
[5] Ashwath, P., Joel, J., Xavior, M.A. and Kumar, H.P., 2018. Effect of SiC and Al₂O₃ particles addition to AA 2900 and AA 2024 MMC’s synthesized through microwave sintering. Materials Today: Proceedings, 5(2), pp.7329-7336.
[6] Sajjadi, S.A., Ezatpour, H.R. and Parizi, M.T., 2012. Comparison of microstructure and mechanical properties of A356 aluminum alloy/Al2O3 composites fabricated by stir and compo-casting processes. *Materials & Design, 34*, pp.106-111.

[7] Gao, S., Wu, Z.S., Jin, P.F. and Wang, J.J., 2011. Study on microstructure and properties of 5A06 aluminum alloy welded joint by deep cryogenic treatment. *In Advanced Materials Research (Vol. 314, pp. 927-931).* Trans Tech Publications.

[8] *International Alloy Designation and Chemical Composition Limit for Wrought Aluminum and Wrought Aluminum*, 2017. registration recorded series, pp3.

[9] Kok, Metin. 2005. Production and mechanical properties of Al2O3 particle-reinforced 2024 aluminium alloy composites. *Journal of Materials Processing Technology* 16, no. 3, pp.381-387.

[10] Bolobov, V. I., and Thanh BinhLe. 2018. Influence of deep cryogenic treatment on structure and wear resistance of materials of hydraulic breaker chisels. In IOP Conference Series: *Materials Science and Engineering*, vol. 327, no. 4, pp. 042016. IOP Publishing.

[11] Raghavendra M.J., Praveen K., Arun R., Arjun S. 2017. A study on microstructure and characterization of aluminum 7075 metal matrix reinforced with silicon carbide particles using stir casting method. *IJRET*, Vol.6, issue 12.

[12] Mazahery, A., Abdizadeh, H. and Baharvandi, H.R., 2009. Development of high- performance A356/nano-Al2O3 composites. *Materials Science and Engineering: A, 518*(1-2), pp.61-64.

[13] Roylance, D., 2001. Stress-strain curves. *Massachusetts Institute of Technology study, Cambridge*.

[14] Ekin, J.W. 2006 Experimental techniques for low temperature measurements *Contemporary Physics, 49*(3), pp.228-229.

[15] Degrieck, J. and Van Paepegem, W., 2001. Fatigue damage modeling of fibre-reinforced composite materials. *Applied mechanics reviews, 54*(4), pp.279-300.