



Enhanced Mechanical and Fatigue Properties In AA5052 Via TiO₂ Nanoparticles Addition Sintering Temperature (ST)

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HIGHLIGHTS

- TiO₂ particles reinforced AA5052 composites are successfully fabricated by the stir casting route.
- The effect of different sintering temperatures (900, 1000 and 1100°C) on fatigue and mechanical characteristics of the metal matrix AA5052 reinforced with 5% of TiO₂ nanoparticles was studied.
- Compared to other sintering temperatures, the nanocomposite with a sintering temperature of 1000°C has the highest hardness, ultimate tensile strength, yield strength and the lowest elongation.

ABSTRACT

The goal of the present work is to study the effect of different sintering temperatures (900, 1000 and 1100°C) on fatigue and mechanical characteristics of the metal matrix AA5052 reinforced with 5% of TiO₂ nanoparticles. The stir casting process is used for manufacturing of AA5052/TiO₂ nanocomposite. The mechanical characteristics of nano composites have been obtained at ambient temperature. The results of mechanical properties showed that the best enhancement in hardness (HB), ultimate tensile strength (UTS) and yield strength (YS) is occurred in nanocomposite with 1000°C sintering temperature (ST). However, the fatigue test results showed that the samples manufactured under 1000°C (ST) have longer fatigue life compared to other materials with different sintering temperatures. The endurance fatigue strength is improved by 7.2% compared to metal matrix. The experimental results showed that the microstructure image of 1000°C (ST) composite has uniformly distributed of TiO₂ in AA5052 matrix.

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1. Introduction

Aluminium matrix composites (AMCs) are well known to be the substances usually reinforced with the matrix by hard particles such as carbides (Al₄C₃, SiC, TiC), oxides (Al₂O₃) or nitrides (TiN, AlN) [1]. Only a few parts of the machine are exposed to static loading, and most parts of the machine are exposed to variable loading. Experimentally, this form of failure is referred to as fatigue when a material is subjected to dynamic loads, it fails at a stress below the yield stress. Fatigue can happen under constant or variable loads. Constant fatigue loading is defined as fatigue under cyclic loading with a consistent amplitude and a constant mean stress or load [2]. Abdizadeh et al. [3] examined the mechanical and microstructure characteristics of AA356 reinforced with different percentages of nano mgo particles. The AA356/mgo were produced by using stir casting at different temperatures of 800, 850, and 950°C and for powder metallurgy at 575, 600, and 625°C. In contrast to the casting samples, they found that powder metallurgy samples show higher porosity sections. Higher porosity portions in sintered composites and more agglomeration and aggregation of mgo nanoparticles in casting samples were seen in scanning electron microscopy images. More homogeneous data and higher values of mechanical properties were expressed in the casting process compared to the powder metallurgy method. T. Rajmohan et al. [4], tested mechanical and microstructural properties of aluminum reinforced with various weight percentages of (CuO) and constant amount of (SiC) using the sintering process. The experimental results showed that the distribution of SiC and CuO in the Al matrix were relatively homogeneous and the mechanical properties were enhanced by increasing the content of CuO reinforcement nanoparticles. In composite,

including (2 CuO+10 SiC wt%), the best improvement was noted. Alalkawi et al. [5] illustrated the effect of adding 10% of alumina Al_2O_3 with a particle size of 50 nm to the AA6061 metal matrix. The AA6061/ Al_2O_3 were manufactured using the stir casting technique. The experimental results showed that the addition of 10wt% Al_2O_3 improved the fatigue life and cumulative fatigue power. The outcomes revealed that the fatigue strength improvement was 12.8% at 10^7 cycles due to 10wt% nano reinforcement. Qusay. K. Mohammed, [6], fabricated AA7075/ Al_2O_3 using the stir casting technique with 6 minutes stirring time and 450 rpm stirring speed. The Al_2O_3 with a particle size of 10 nanometer and different weight percentages of (0.3, 0.5, and 0.7wt%) were used to reinforced AA7075. The outcomes showed that the best improvement in fatigue life and strength occurred at 0.3% of Al_2O_3 . M. Vaghari et al. [7] used wet attrition milling followed by hot forward extrusion process for fabricating Al reinforced with alumina Al_2O_3 with different weight percentages of (4, 6 and 8%). The findings showed that the existence of up to 6% of Al_2O_3 nanoparticles improved the fatigue strength of Al/ Al_2O_3 nanocomposites, while the greater amount of reinforcement has a negative impact on the strength of the fatigue. Al-Alkawi et al. [8] studied the effect of 6% of alumina Al_2O_3 nanoparticles with AA7100 metal matrix on constant fatigue strength and life at 200°C under rotating bending loading. The outcomes showed that the fatigue strength improvement factor was obtained to be from 0.84 to 4.84 for 10^3 and 10^5 cycles, respectively. However, increasing the life from 10^3 and 10^5 cycles leads to an increase in FSIF of 4. On the other hand, the FSIF at 200°C was obtained to be 0.11. Abdulridah et al. [9] investigated the effect of cryogenic temperature on mechanical properties and fatigue behavior of AA2024/ Al_2O_3 nanocomposites. The stir casting technique was used to fabricate the AA2024/ Al_2O_3 with different weight percentages of Al_2O_3 . It was found that the composite that contains 0.9% Al_2O_3 showed the best properties and maximum enhancement, which shows an improvement in ultimate strength, yield stress and Brinell hardness of 9.9, 14.63 and 14.28%, respectively compared to the base metal matrix. The fatigue endurance limit of the 0.9wt% Al_2O_3 composite was taken at 10^7 cycles in two cases without (CT) and with (CT). At 10^7 cycles, the exhaustion limit rises from 83 MPa without (CT) to 89.6 MPa with (CT) resulted in an increase in the fatigue limit of 7.95%. Mamoon A. A. Al-Jaafari [10] studied the fatigue behavior of AA6061 metal matrix reinforced with SiC of (10nm) particle size and with different weight percentages of SiC. The AA6061/SiC nanocomposites were fabricated using stir casting method with rotating bending type of load at room temperature and with a stress ratio of (R=-1). At, 2wt%, the highest fatigue strength and life were made. Due to relatively uniform distribution strengthening of nano particles and limited porosity, percent nano particles were obtained. The percentage of rise in endurance fatigue limits for 10^7 and $5 * 10^8$ cycles were 11.48% and 11.05%, respectively. Iman S. El-Mahallawi et al. [11] tested the mechanical properties of A356 reinforced with alumina (Al_2O_3), titanium dioxide (TiO_2) and zirconia (ZrO_2) nano-particles (40 nm). They were stirred in the A356 matrix at variable stirring speeds varying from (270, 800, 1500, and 2150 rpm) in both the semisolid (600 °C) and liquid (700 °C) states with a constant stirring time of one minute with different fraction ratios ranging from (0 –5 %) by weight. The experimental results showed that the mechanical properties (strength, elongation, and hardness) of Al_2O_3 , TiO_2 and ZrO_2 nanoreinforced castings made in the semi-solid state (600 °C) with 2 wt.% Al_2O_3 and 3 wt.% TiO_2 or ZrO_2 at 1500 rpm stirring speed were improved. Ali Y. Khenyab et al. [12] manufactured AA7075-T61 metal matrix with different weight percentages of Al_2O_3 (10%,15%, and 20%) and TiO_2 with 5% were used as reinforcement materials using the stir casting technique. The results revealed improving in fatigue life and mechanical properties. It has been noticed that the well distribution of nano particles on the matrix in microstructure examination, leads to reduce the grain size which leads to improving in fatigue life higher than the base metal and other composites by (3.5%, 7.7% and 9.7%). The objective of the this work is to enhance the mechanical and fatigue properties of AA5052 reinforced with TiO_2 nanoparticles using three sintering temperature of (900, 1000 and 1100 °C). Some of the previous studies used different weight percentages of nanoparticles and variable stirring speed and they also used various temperatures using two methods, namely stir casting and powder metallurgy and they found improvement in mechanical and microstructure properties, but the current study used constant weight percentage of TiO_2 nanoparticles with various sintering temperatures of (900, 1000 and 1100 °C) using the stir casting method. The results of this study revealed an improvement in mechanical and fatigue properties at a sintering temperature of (1000 °C).

2. Experimental work

The chemical composition of AA5052, sample preparation and standard geometry of the samples are presented in this section.

2.1 Material

Aluminum metal matrix AA5052 is used in the present work. AA 5052 is one of the Al-Mg alloys that, due to its strong properties, is mainly used in the automotive, aerospace and marine industries [13-16]. AA5052 is enhanced with TiO_2 with a particle size of (30nm). The chemical composition of AA5052 is as shown in Table I.

2.2 Manufacturing of specimens

The AA5052/ TiO_2 were fabricated by using the stir casting method. The procedure of the process can be as follows [17]:

- 1) Initially, the desired sample weight is determined by the rule of volume x density

$$m=V \times \rho \rightarrow m=0.6 \times 12 \times 5 \times 3 \rightarrow m=108 \text{ gm} \quad (1)$$

Where (m) is mass in (gram), (V) is the volume in cm^3 and (ρ) is density in $g/cm^3=3$, (h) thick in $cm=0.6$, (L) length in $cm=12$ and (b) wide in $cm=5$.

- 2) Calculate the proportions of added elements (magnesium and chromium).
 $\text{Mg weight} = 108 \times 0.0317 \rightarrow \text{Mg weight} = 3.4236\text{g}$
 $\text{Cr weight} = 108 \times 0.0043 \rightarrow \text{Cr weight} = 0.4644\text{g}$
 $\text{Al weight} = 3.4236 - 0.4644 \rightarrow \text{Al weight} = 2.9592\text{g}$
- 3) After that, put the aluminum, which is in the form of pure wires, in the crucible and then start fusion it up to 750°C for 5 minutes by using a gas furnace. During the aluminum smelting process, a sand mold is prepared according to the dimensions of the sample.
- 4) Chromium is added to the melt, which is placed in aluminum foils, wrap it well and then immerse it in the molten metal. Leave it about three minutes, then mixing is done by using an electric mixer at a speed of 600 r.p.m for a minute.
- 5) After mixing is completed, magnesium is added which is in the form of strips and cover it with aluminum foils, then expel the air, then add it to the inside of the molten, keeping it under the slag in the melt as well. Then, mix it until the magnesium and chromium are distributed well, then add 1 gram of aqueous aluminum chloride (AlCl_3) for the purpose of removing slag and expelling gases. After that, the slag is removed and the first casting is poured without adding nano titanium oxide after it has frozen and then, remove it from the mold.
- 6) For the second, third and fourth castings, the same previous steps are used, but nano titanium oxide is added after coating it with aluminum foil to expel the gases before adding the magnesium. It is mixed for five minutes using an electric mixer, then magnesium is added in the same way as the previous one also mix well and aluminum chloride is added as an aid to remove slag. Then, leave it for 10 Minutes until the sample freezes, after that it is removed from the sample. Subsequently, the mixture is poured into the mold and according to the temperature, where the second casting was added at a temperature of 900, the third casting at a temperature of 1000 and the fourth at a temperature of 1100.

2.3 Tensile test

In order to determine the mechanical behavior of cast AA5052 and fabricated nanocomposites, tensile tests were performed. A tensile test specimen according to ASTM D 638-97 [18] is shown in Figure 1. The tensile samples were manufactured from the cast AMCs. Specimens have been tested using a tensile machine at room temperature.

2.4 Fatigue test

The samples were manufactured according to D 3479/D 3479M-96 ASTM [19]. In order to satisfy the machine test section that is appropriate for flat plate specimens, fatigue specimens were cut to acceptable dimensions. Figure 2 demonstrates the shape and dimensions of the fatigue sample [20]. The AVERY fatigue testing machine of type-7305 was used to conduct all the fatigue testing.

The AVERY fatigue testing machine of type-7305 was used to apply reverse loads. For the bend test, grips are provided where the load is imposed by an oscillating spindle driven by a connecting rod, crank, and double eccentric attachment at one end of the specimen. To give the required range of bending angle, the eccentric attachment is adjustable. From the deflection, the applied stress is measured. The motor is equipped with a revolution counter to record the number of cycles. The cycling rate is 1400 rpm [20]. The stress ratio of $R = -1$ was adjusted for the device. On each collection of specimens, a series of experiments were carried out by altering the deflection angle each time and recording the number of cycles to failure. Figure 3 shows an example of the specimen after failure of the fatigue test.

Table 1: Chemical composition of AA5052 alloy.

Alloy	Mg	Cr	Cu	Fe	Mn	Si	Zn	Other elements		
								Each	Sum	Al
<i>Standard [16]</i>	2.2- 2.8	0.15-0.35	0.1	0.4	0.1	0.25	0.1	0.05	0.15	Balance
<i>Experimental</i>	2.6	0.29	0.09	0.33	0.08	0.24	0.1			Balance

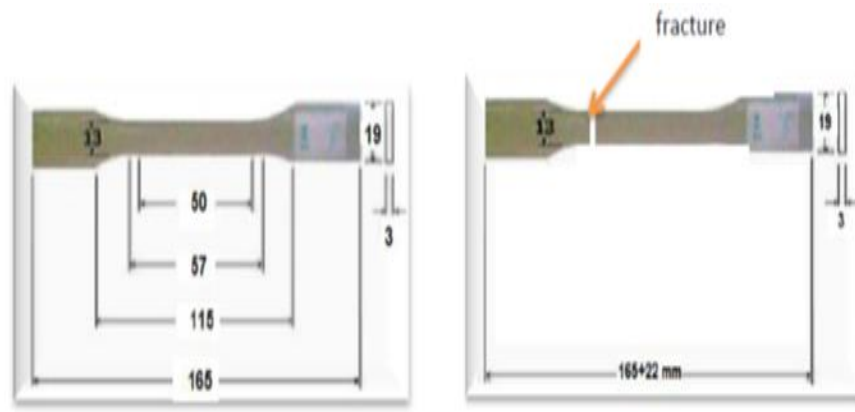


Figure 1: Tensile test specimen with dimensions (a) before the test and (b) after the test for room temperature (all dimensions are in mm) [18].

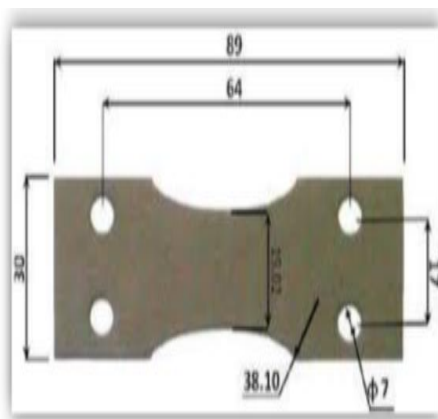


Figure 2: Fatigue specimens before the test (all dimensions are in mm) with a thickness of (4mm) according to D 3479/D 3479M-96 ASTM [19].

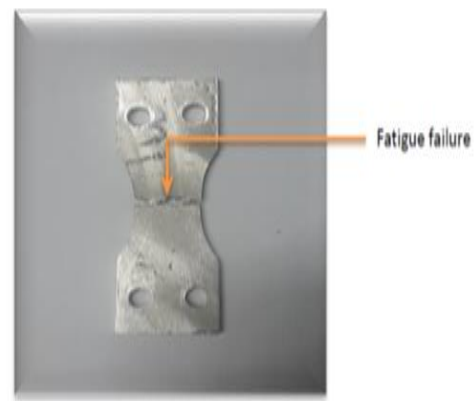


Figure 3: Fatigue failure (after test) of Aluminum metal matrix nanocomposite.

3. Results and discussion

3.1 Tensile test results

For the evaluation of ultimate tensile strength, yield tensile strength and other properties, tensile tests were used. The experimental test established stress-strain curves for pure AA5052 and AA5052 reinforced with constant weight percentage of (5wt%) of TiO_2 nanoparticles, as shown in Figure 4. The results of the mechanical properties of pure AA 5052 and its composites with various sintering temperatures calculated from Figure 4 are shown in Table II. Jufu Jiang et al. [21] tested mechanical properties of AA2024/ Al_2O_3 by using ultrasonic assisted semisolid stirring (UASS) method and rheoformed into a cylinder component. The outcomes showed that at the bottom of the rheoformed composite components, the optimum ultimate tensile strength (UTS) of 358 MPa and YS of 245 MPa was obtained after 25-min stirring of the composite semisolid slurry with 5% Al_2O_3 nanoparticles at 620 °C.

Table II shows that the mechanical properties of nanocomposite were affected by the sintering temperature. It can be inferred that nanocomposite AA5052/ TiO_2 at 1000(ST) has the best mechanical properties. The above improvements may be coming from the existence in the matrix of hard TiO_2 particles and the low degree of porosity and uniform distribution of the nano particles. The introduction of TiO_2 nanoparticles into the aluminum matrix is believed to provide some heterogeneous nucleation sites during sollicitation resulting in refined grains [22, 23] and the sintering temperature, which leads to high dislocation and this will reduce the grain size.

3.2 Fatigue S-N curve results

The specimens were tested under constant amplitude fatigue. The results of this test are illustrated in Tables III and IV. The applied stress can be calculated from the following equation [24]:

$$\sigma = \frac{0.15Eh\delta}{L_0^2} \quad (2)$$

Where σ is applied stress in (MPa), modulus of elasticity is E in (Gpa)=70 Gpa, h is thickness of specimen=4mm, maximum deflection is δ in (mm) and L_0 is the effective length in (mm). The effective length can be calculated from the equation [24]:

$$L_0 = \frac{4L^2 - 2.465\delta^2}{4L} \quad (3)$$

Where L is half length = 32mm, deflection is calculated from the calibration curve [24], (84) specimens were tested to get the S-N curve, (21) specimen for condition as received AA5052 and other samples for conditions with 5wt%TiO₂ and different sintering temperatures. The S-N curve was plotted depending on fatigue equations, as shown in Figure 5.

The (S-N) curves equations are calculated according to Basquin equation of the form of $\sigma = A [(Nf)]^b$ where A and b are material constant fatigue parameters and endurance limit, as shown in Table IV.

Table 2: Tensile test results for metal matrix and nanocomposites at different sintering temperatures.

Condition	UTS (MPa)	YS (MPa)	Elongation (%)	HB
AA5052 metal matrix	200	105	13.4	45
Nanocomposite 5wt%TiO ₂ (900ST)	217	114	13.0	47
Nanocomposite 5wt%TiO ₂ (1000ST)	244	128	12	51
Nanocomposite 5wt%TiO ₂ (1100ST)	225	121	12.5	49

Table 3: S-N results for different sintering temperatures.

Specimen No.	Theta (degree)	Applied stress (MPa)	No. of cycles to failure(Nf)	Average no. of cycles (Nfav)	Condition
1,2,3	20°	285.4747	400,500,450	450	As received(AR) AA5052
4,5,6	18°	252.4594	600,700,650	650	
7,8,9	16°	222.6574	1200,1400,1300	1300	
10,11,12	14°	193.5704	4000,4200,4100	4100	
13,14,15	12°	167.2690	16000,17100,16550	16550	
16,17,18	10°	132.8827	29000,31800,30400	30400	
19,20,21	8°	107.5137	72600,61800,67200	67200	
22,23,24	20°	285.4747	600,800,700	700	900(ST) 5wt%TiO ₂ nanocomposite
25,26,27	18°	252.4594	1000,1300,1150	1150	
28,29,30	16°	222.6574	1800,2000,1900	1900	
31,32,33	14°	193.5704	3400,5500,4450	4450	
34,35,36	12°	167.2690	20000,21500,20750	20750	
37,38,39	10°	132.8827	38800,40600,39700	39700	
40,41,42	8°	107.5137	96400,101200,98800	98800	
43,44,45	20°	285.4747	800,1000,900	900	1000(ST) 5wt%TiO ₂ nanocomposite
46,47,48	18°	252.4594	1200,1600,1400	1400	
49,50,51	16°	222.6574	2400,3000,2700	2700	
52,53,54	14°	193.5704	4500,6000,5250	5250	
55,56,57	12°	167.2690	30700,29800,30250	30250	
58,59,60	10°	132.8827	48600,51800,50200	50200	
61,62,63	8°	107.5137	127600,134000,130800	130800	
64,65,66	20°	285.4747	700,800,750	750	1100(ST) 5wt%TiO ₂ nanocomposite
67,68,69	18°	252.4594	1100,1400,1250	1250	
70,71,72	16°	222.6574	2000,2400,2200	2200	
73,74,75	14°	193.5704	3600,4800,4200	4200	
76,77,78	12°	167.2690	20100,19100,19600	19600	
79,80,81	10°	132.8827	35500,36800,36150	36150	
82,83,84	8°	107.5137	88000,78800,83400	83400	

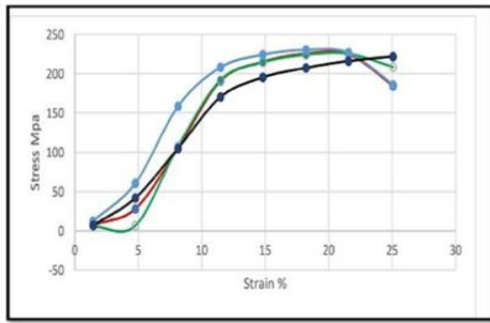


Figure 4: Stress-strain curve of metal matrix and nanocomposites at various sintering temperatures (ST).

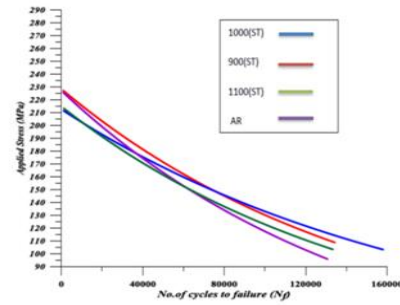


Figure 5: S-N curve for different sintering temperatures of (900, 1000 and 1100).

Table 4: Fatigue parameters for different sintering temperatures.

Condition	A	b	Basquin equation	Endurance limit (MPa)
AR	796	-0.1739	$\sigma = 796Nf^{-0.1739}$	48.26
900(ST)	896	-0.1807	$\sigma = 896Nf^{-0.1807}$	48.68
1000(ST)	919	-0.1781	$\sigma = 919Nf^{-0.1781}$	52.07
1100(ST)	974	-0.1904	$\sigma = 974Nf^{-0.1904}$	45.26

The results of endurance limit shows that the higher fatigue strength occurred for the nanocomposite under 1000 °C (ST) then when the sintering temperature increases to 1100°C the endurance limit decreases due to less bonding between matrix and nanoparticles and less distribution of TiO₂ compared to other nanocomposites. Using Basquin's formula, it is easy to express the fatigue strength in terms of the stresses corresponding to a given lifetime on the mean S-N curve. The results of the fatigue strength are shown in Figure 6. It is clear that the endurance fatigue limit of 1000°C (ST) nanocomposite is higher than the matrix by 7.2%.

3.3 Microstructure

The distribution of the nanoparticles in the matrix plays a significant role in determining the mechanical properties. If there are fewer porosities after casting, the nanocomposite gets excellent characteristics. The pure AA 5052 and AA5052/TiO₂-5wt% at 1000°C sintering temperature (of the third) composite samples were carried out with optical micrographs. Figure 7 shows the optical micrographs of the base metal and 5wt% TiO₂ nanocomposite at 1000°C (ST). Figure 7(b) reveals the presence of TiO₂ with homogenous dispersion of TiO₂ into the base metal. Also, the microstructure of nanocomposite reveals small discontinuity and a reasonably uniform distribution of TiO₂ particles in the aluminum matrix. Due to the nanosize particles and sintering temperature, the higher fatigue intensity increases and the surface morphology is shown in Figure 7. The density of dislocation and the minimization of grain size defects contribute to an improvement in the mechanical properties and strength of fatigue.

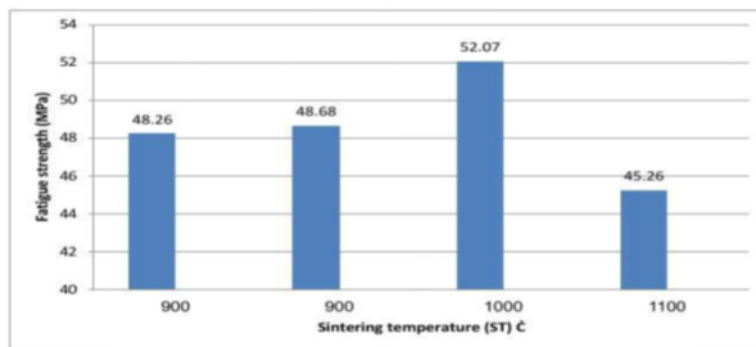


Figure 6: Fatigue strength at various sintering temperatures.

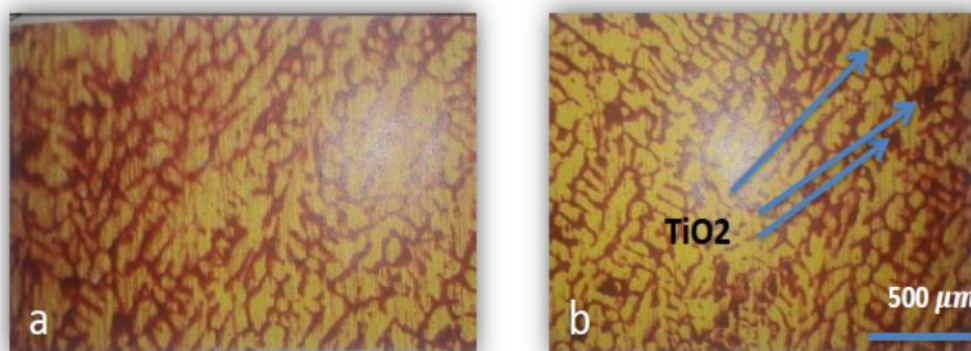


Figure 7: Optical micrographs of as-received and nanocomposite at 1000°C, at (a) Zero nano (As-received) AA5052, (b) 5wt% TiO₂ nanocomposite at 1000°C (ST).

4. Conclusions

The objective of this work is to enhance the mechanical and fatigue properties of AA5052 reinforced with TiO₂ nanoparticles using three sintering temperatures of (900, 1000 and 1100 °C). The main problem in the present study is the manufacturing of nanocomposite in a high sintering temperature of (1100 °C) because this leads to evaporate the alloys, which leads to generate oxides. The major conclusions extracted from the present work can be summarized as follows:

- 1) TiO₂ particles reinforced AA5052 composites are successfully fabricated by the stir casting route.
- 2) It has been also shown that the sintering temperature of 1000°C with 5wt% TiO₂ nanocomposites has better mechanical, hardness and fatigue properties than other sintering temperatures of (900 and 1100°C) and has a major effect on the above characteristics.
- 3) Compared to other sintering temperatures, the nanocomposite with a sintering temperature of 1000°C has the highest hardness, ultimate tensile strength, yield strength and the lowest elongation.
- 4) Compared to the other manufactured nanocomposites and base metal, the nanocomposite AA5052 supported with 5wt% TiO₂ with 1000°C (ST) showed high strength and fatigue life.
- 5) The improvement of mechanical and fatigue properties is attributed to the grain refinement and uniformly distribution of TiO₂ particles into a metal matrix.

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Author contribution

All authors contributed equally to this work.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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