American Journal of Energy Engineering
2023; 11(2): 67-70
http://www.sciencepublishinggroup.com/j/ajee
doi: 10.11648/j.ajee.20231102.15
ISSN: 2329-1648 (Print); ISSN: 2329-163X (Online)

Effect of Heat Treatment on Mechanical Properties of Graphene Reinforced Titanium Matrix Composite Materials

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To cite this article:
Xinlong Jiao, Zhifang Cheng, Hongmei Zhang, Zhenyu Wei, Luye Wei, Juntong Ren, Siwei Wang, Yongfan Li. Effect of Heat Treatment on Mechanical Properties of Graphene Reinforced Titanium Matrix Composite Materials. *American Journal of Energy Engineering*. Vol. 11, No. 2, 2023, pp. 67-70. doi: 10.11648/j.ajee.20231102.15

Received: April 29, 2023; **Accepted**: June 1, 2023; **Published**: June 5, 2023

Abstract: Graphene has a significant reinforcing effect. Titanium and titanium alloys have excellent properties such as high specific strength and good corrosion resistance compared to steel, and have broad application prospects in fields such as aerospace, automotive manufacturing, chemical engineering, and biomedical equipment. Graphene, as a new member of the carbon family, not only has excellent physical and mechanical properties, but also has a unique two-dimensional structure. This 2D structure has a higher specific surface area than CNT or graphite. Thus providing more contact area and interaction with the matrix material, these characteristics are very attractive for the research of composite materials. MLG/Flake Ti hybrid powder was prepared using high-energy ball milling, and then MLG/Flake Ti composite material was prepared through low-temperature high-pressure SPS preforming and high-temperature heat treatment (950°C). Perform high-temperature heat treatment at 950°C and set the insulation time to 10 minutes and 60 minutes respectively. After heat treatment, the hardness and yield strength of the material were significantly improved. After 10 minutes of heat treatment, the sample with 0.2wt% MLG added showed a yield strength of 2225MPa and an elongation of 30%, demonstrating a good match between strength and plasticity. Compared with the sample with only 0.1wt% MLG added, the strength improvement is nearly 60%. This indicates that the increase in MLG content has a significant strengthening effect.

Keywords: Graphene, Titanium Based, Strength, Hardness, Heat Treatment

1. Introduction

As a new two-dimensional carbon nano material, graphene (MLG for short) has high elastic modulus and fracture strength, which makes it an ideal nano reinforcement phase for lightweight metal matrix composite. Researchers studied MLG reinforced composite materials such as Al, Mg, Ni, Cu, Fe [1-7]. These studies indicate that graphene has a significant enhancing effect. Titanium and titanium alloys have excellent properties such as high specific strength and good corrosion resistance compared to steel, and have broad application prospects in fields such as aerospace, automotive manufacturing, chemical engineering, and biomedical equipment. Mu X N, et al. [8-10] Multilayer graphene (9-10 layers)/pure titanium (titanium powder average diameter 0-35μm) prepared by ball milling, spark plasma sintering (SPS, sintering temperature 876K), and subsequent hot rolling (HR, temperature 1223K). Three strengthening mechanisms were analyzed for composite materials: load transfer, fine grain strengthening, and texture strengthening.

Huang H, et al. [11, 12] used a sheet-like powder metallurgy process to achieve uniform adhesion of CNTs to the surface of 6061Al powder through low-speed long-term ball milling (135rmp, 8h), and then achieved embedding of CNTs onto the surface of the powder through high-energy ball milling at 270rmp, 1h. Subsequently, a uniformly distributed structure of CNT was obtained through processes such as annealing, sintering, and rolling. Zhao M, et al. and others mixed three different sizes of graphite oxide (S-rGO, M-rGO, L-rGO) with flake Al powder, and then hot pressed and sintered to obtain nano laminated structure [13, 14].

2. Experimental Method

The titanium powder used is commercial grade pure titanium. The purity of the powder is greater than 99%, with a particle size distribution of 0-45 μ m. The original graphene powder agglomerates severely and is mainly dispersed by ultrasound. First, use the Analytical balance to weigh the MLG with a specific mass fraction (0.1wt%, 0.2wt%), then transfer it to a beaker, add an appropriate amount of anhydrous ethanol, and put it into the ultrasonic cleaner for more than 30 minutes. Subsequently, the MLG was uniformly adhered to the surface of the grinding ball through low-speed ball milling.

The SPS-3.20-MV discharge plasma sintering system is used, with an impact current on/off ratio of 12:2 and a pulse current cycle of approximately 3.3ms. During the sintering process, the heating rate, sintering temperature, and holding time are adjusted by manually controlling the current and voltage.

In order to avoid the reaction between MLG and Cp-Ti, and to make the structure as dense as possible, a high-pressure and low-temperature sintering scheme is adopted. The sintering temperature used is 600°C, the pressure is 300MPa, the heating rate is 100°C/min, and the insulation is 5 minutes. After taking out the sample, the graphite paper and the reaction layer between the graphite paper and Ti on the surface of the sample are removed by turning.

Use a vacuum heat treatment furnace for heat treatment. Due to the small size of the sample undergoing heat treatment, the method of heating the sample into the furnace is adopted. The sample is directly placed in a heat treatment furnace at 950°C, with a holding time of 10 minutes and 60 minutes. After removing the sample, it is quenched in cold water.

Characterization of material hardness using Vickers hardness. The compression performance is tested by universal testing machine. The compressed sample is A small cylinder of φ4X4. Constant displacement loading, with a loading rate of 1mm/min.

3. Experimental Results and Discussion

3.1. Mechanical Properties Before Heat Treatment

The microstructure after SPS sintering exhibits the characteristics of debris forming interface coating titanium sheets, and MLG covering or inserting into the surface of titanium sheets. The addition of MLG can significantly improve the yield strength of the billet, while also bringing about plastic loss. As the number of layers increases, the strengthening effect of MLG weakens.

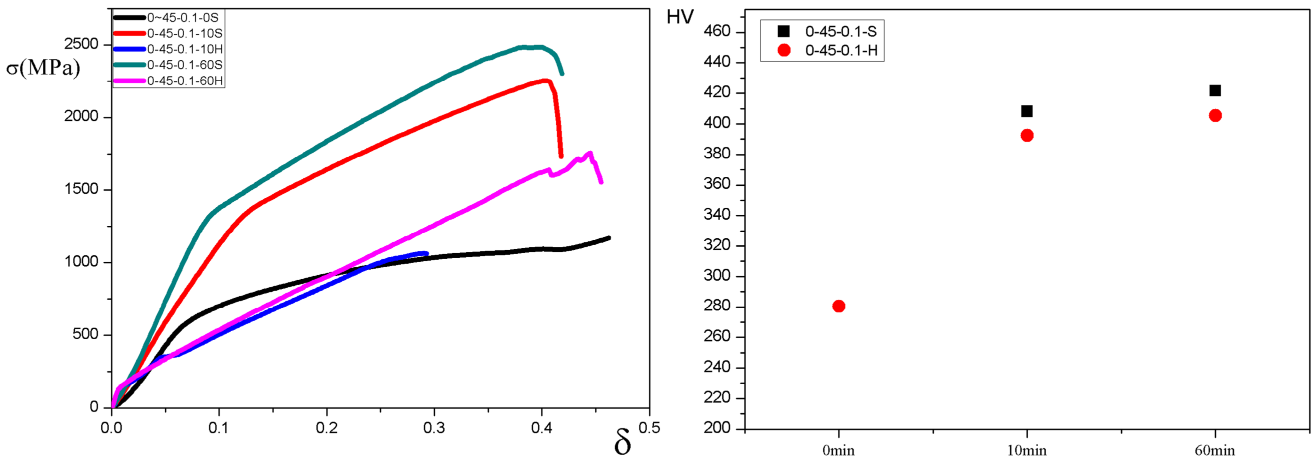
Table 1 shows the compression properties and hardness of SPS preformed materials. The composite material with 0.1wt% MLG added has a yield strength of 618MPa. The elongation is high at 36%. When the content of MLG is 0.2wt%, the mechanical properties of the material are significantly improved. The yield strength increased by 150%, while the elongation decreased. The increase in graphene content showed a significant strengthening effect on the material, achieving a yield strength of 1533Mpa.

Table 1. *Compressive strength and hardness.*

Particle size (μm)	0~45	0~45
Component (wt%)	0.1	0.2
compressive strength (Mpa)	618	1533
Elongation rate (%)	36	22
Vickers hardness (HV)	280	230

3.2. The Effect of Heat Treatment Time on Mechanical Properties

With the prolongation of heat treatment time, there is a certain trend of grain growth, but due to the distribution of MLG at the interface, grain growth is hindered, and the trend of grain growth is not significant, with a relatively uniform size. The interface becomes denser, the bonding becomes better, and the gap decreases.



(a) compressive strength (b) hardness

Figure 1. *Mechanical properties after heat treatment.*

Figure 1 shows the compressive mechanical properties of 0-45 μm-0.1wt% MLG can be seen from the graph that the yield in all S directions is significantly higher than that in the H direction. After heat treatment, the yield strength in the S direction is maintained at approximately 1400Mpa, while in the H direction it is maintained at approximately 140Mpa. The strength in the S direction is about 10 times that of the H direction.

Figures 1-b shows The hardness of 0-45 μm-0.1wt% MLG material after heat treatment, which can be significantly improved from 280HV before heat treatment to 392HV (10min) and 405HV (60min), with an increase of nearly 30% from the H-direction. This corresponds to the significant increase in yield strength of the material through heat treatment. After heat treatment, the hardness in the S direction is 408HV (10min) and 421HV (60min), respectively, with the hardness in the S direction slightly higher than that in the H direction. Extending the heat treatment time results in minimal tissue change and less significant hardness improvement, which is also consistent with the results of compression experiments.

3.3. Effect of MLG Content on Mechanical Properties After Heat Treatment

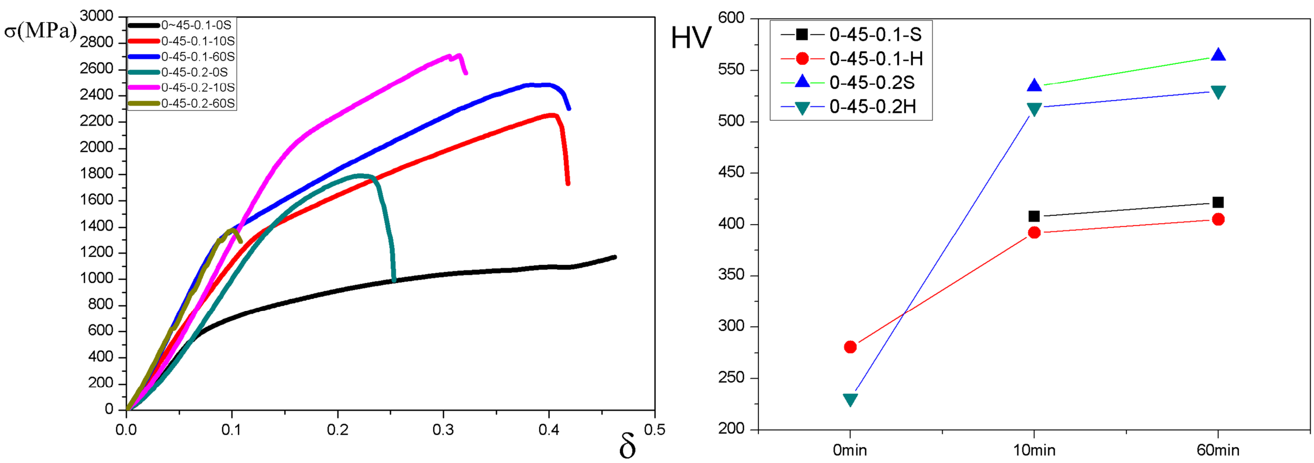
G ü rb ü z M and Mutuk T prepared graphene (5-10nm thick, 5-10 nm thick) through ultrasound, ball milling, cold pressing forming, and high-temperature vacuum sintering μ M wide) reinforced titanium (titanium powder, 70 μ m) The effects of sintering time, temperature, and graphene content on the hardness and microstructure of composite materials were systematically studied. The Vickers hardness of composite materials first increases and then decreases with the content of graphene, and increases with the increase of sintering temperature. 0.15wt% MLG-Ti achieved the highest hardness of 552VH during insulation at 1100°C for 60 minutes. The literature points out that uniformly dispersed MLG and good interface binding play a reinforcing role. The increase in temperature accelerates the densification process, while the large surface of MLG effectively prevents grain growth [15].

Figure 2-a shows the compressive mechanical properties of the material after heat treatment. When the MLG content increased from 0.1wt% to 0.2wt%, the yield strength of the preformed material significantly increased, from 618MPa to 1589MPa, an increase of nearly 2.5 times, but the elongation decreased from 36% to 22%. After 10 minutes of heat treatment, the sample with 0.2wt% MLG added showed a yield strength of 2225MPa and an elongation of 30%, demonstrating a good strength plasticity match. Compared with materials with only 0.1wt% MLG added, the strength improvement is nearly 60%. This indicates that the increase in MLG content has a significant strengthening effect.

When the heat treatment time was extended to 60 minutes, the elongation of the sample with the addition of 0.2wt% MLG was only 9%, showing strong brittleness. At the same time, the yield strength decreased to 1377 MPa. When observing the microstructure, it was found that there was too much MLG enrichment at the interface and prolonged heat treatment, resulting in material embrittlement.

From the H direction in Figure 2-a, the addition of 0.1wt% MLG and 0.2wt% MLG significantly improved the hardness value of the material after heat treatment. After heat treatment, the hardness of the material with the addition of 0.2wt% MLG increased from 230HV before heat treatment to 514HV (10min) and 530HV (60min), with an increase of nearly 120%. Compared with the material with only 0.1wt% MLG added, the hardness value of the material after heat treatment significantly increased when the MLG content increased to 0.2wt%, from around 410HV at low content to 550HV.

After heat treatment, the difference in hardness between the H and S directions is very small, and the hardness in the S direction is slightly higher than that in the H direction. As the heat treatment time increases from 10 minutes to 60 minutes, the increase in hardness is not very significant.



(a) compressive strength (b) hardness

Figure 2. *Mechanical properties after heat treatment with different MLG contents.*

4. Conclusion

1) After heat treatment of graphene reinforced titanium matrix composites, their hardness and yield strength can be significantly improved. The addition of 0.1wt% MLG graphene reinforced titanium matrix composite material significantly improved the yield strength and elongation of the material with the prolongation of heat treatment time. After 60 minutes of heat treatment, the yield strength and elongation of the material were 1450MPa and 38%, respectively.
2) After heat treatment, the hardness increases and the insulation time is extended, resulting in a smaller increase in hardness. At the same time, materials with high content of MLG showed significantly higher hardness than samples with low content. After heat treatment with the addition of 0.1wt% MLG and 0.2wt% MLG, the hardness values in the H direction were significantly improved. After heat treatment, the difference in hardness between the H and S directions is very small.

Acknowledgements

This paper is supported by funding from the College Student Innovation and Entrepreneurship Training Program.

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