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Thermal Stability Analysis of Az91-Cnt Magnesium Nanocomposites Under Controlled Thermal Exposure

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ABSTRACT

Thermal stability is a critical requirement for magnesium-based materials intended for applications involving prolonged exposure to elevated temperatures. However, conventional thermal characterisation methods mainly focus on identifying phase transformation temperatures and provide limited information on time-dependent microstructural and mechanical stability under service-like conditions. Therefore, this study aims to evaluate the thermal stability of AZ91 magnesium alloy reinforced with carbon nanotubes through a stability-oriented assessment framework. A comprehensive Thermal Stability Analysis was employed to investigate the behaviour of AZ91–CNT magnesium nanocomposites subjected to controlled thermal exposure. Based on critical thermal events identified from thermal analysis, both reinforced and unreinforced samples were exposed to selected isothermal temperatures, followed by systematic evaluation of microstructural evolution and hardness retention. The results demonstrate that carbon nanotube reinforcement significantly enhances the thermal stability of AZ91 magnesium alloy by delaying the dissolution and coarsening of the β-$Mg_{17}Al_{12}$ intermetallic phase. The AZ91-CNT nanocomposites exhibited minimal hardness degradation and preserved microstructural features when exposed to temperatures within the range of 150-200 °C, whereas the unreinforced AZ91 alloy showed earlier onset of thermal softening and microstructural instability. The findings reveal a clear extension of the stable operating temperature window for the CNT-reinforced alloy compared to the unreinforced counterpart. This enhanced thermal stability is attributed to the role of carbon nanotubes as diffusion barriers and microstructural pinning sites that restrict phase instability during prolonged thermal exposure. In conclusion, the Thermal Stability Analysis approach provides a practical and service-relevant evaluation of material performance and establishes reliable thermal operating limits for AZ91-CNT magnesium nanocomposites intended for temperature-sensitive engineering applications.

Keywords:

Thermal stability analysis; AZ91 magnesium alloy; carbon nanotubes; thermal exposure; phase stability

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1. Introduction

The increasing demand for lightweight structural materials capable of maintaining mechanical integrity under elevated temperatures has intensified research on magnesium alloys for automotive, aerospace, and energy-related applications. Owing to their low density and high specific strength, magnesium alloys offer significant potential for weight reduction and improved fuel efficiency. However, their relatively poor thermal stability during prolonged exposure to intermediate temperatures remains a critical limitation for wider engineering adoption [1,2].

Among commercially available magnesium alloys, AZ91 is one of the most widely utilised due to its favourable combination of cast ability, strength, and corrosion resistance [3]. Nevertheless, the thermal stability of AZ91 alloy is strongly governed by the behaviour of the β-$Mg_{17}Al_{12}$ intermetallic phase, which tends to dissolve, coarsen, and redistribute at temperatures above approximately 150 °C. Such microstructural instability accelerates thermal softening, reduces hardness, and degrades load-bearing capability, thereby restricting the usable service temperature range of AZ91 alloy [4,5]. Previous studies have demonstrated that prolonged thermal exposure significantly influences aluminium diffusion and β-phase instability, leading to deterioration of mechanical performance at elevated temperatures [6,7].

To address these limitations, extensive research has been conducted to enhance the thermal and mechanical performance of magnesium alloys through microstructural modification and reinforcement strategies. In recent years, carbon nanotubes have emerged as promising reinforcements for magnesium-based composites due to their exceptional mechanical strength, high thermal stability, and large aspect ratio. Numerous studies have reported that CNT incorporation improves hardness, strength, and wear resistance of magnesium alloys through mechanisms such as load transfer, grain refinement, and dislocation pinning [8–10]. Beyond mechanical enhancement, CNTs have also been suggested to improve thermal stability by acting as diffusion barriers and microstructural pinning sites that restrict grain boundary migration and phase coarsening during thermal exposure [11].

Despite these reported benefits, the thermal exposure behaviour and long-term stability limits of CNT-reinforced AZ91 magnesium composites remain insufficiently understood. Thermal characterisation of magnesium alloys has traditionally relied on Differential Scanning Calorimetry to identify phase transformation temperatures and thermodynamic events during continuous heating [12,13]. While DSC provides valuable insights into transformation behaviour, it does not adequately capture time-dependent microstructural degradation and mechanical property retention under sustained thermal exposure. Consequently, reliance on DSC alone may lead to an overestimation of safe operating temperatures for magnesium alloys and their composites [14].

Thermal Stability Analysis offers a complementary and application-oriented approach by systematically evaluating microstructural stability and mechanical performance following controlled isothermal thermal exposure. By correlating exposure temperature with phase stability and hardness evolution, TSA enables the identification of practical stability windows and degradation onset under service-relevant conditions. However, TSA-based investigations focusing on CNT-reinforced AZ91 magnesium alloys are scarce, and systematic frameworks integrating DSC-informed temperature selection with post-exposure microstructural and mechanical assessment have not been sufficiently established in existing literature.

Therefore, the present study aims to perform a comprehensive Thermal Stability Analysis of AZ91–CNT magnesium nanocomposites subjected to controlled thermal exposure. By integrating DSC-informed temperature selection with systematic evaluation of microstructural evolution and hardness retention, this work seeks to establish a stability-oriented assessment framework, quantify the role of CNT reinforcement in delaying β-$Mg_{17}Al_{12}$ phase instability, and define practical operating temperature limits for AZ91–CNT nanocomposites under prolonged thermal exposure. The findings are expected to provide fundamental insight into thermal stabilisation mechanisms in magnesium-based nanocomposites and to support their application in temperature-sensitive engineering components.

2. Methodology

2.1 Materials and Composite Fabrication

Commercial AZ91 magnesium alloy powder was used as the matrix material in this study, owing to its widespread industrial application and well-documented thermal behaviour [15]. Multi-walled carbon nanotubes were employed as the reinforcement material due to their excellent mechanical properties, high thermal stability, and suitability for magnesium-based nanocomposites [16].

The AZ91–CNT nanocomposites were fabricated using a powder metallurgy route to promote uniform dispersion of carbon nanotubes within the magnesium matrix and to minimise CNT agglomeration, which is critical for achieving consistent thermal stability behaviour [17]. Unreinforced AZ91 samples were prepared using identical processing conditions, excluding the addition of CNTs, to serve as reference materials and to isolate the effect of CNT reinforcement on thermal stability.

2.2 Controlled Thermal Exposure Procedure

Thermal Stability Analysis was carried out by subjecting both unreinforced AZ91 alloy and AZ91–CNT nanocomposites to controlled isothermal thermal exposure at selected temperatures of 150, 175, 200, 225, and 250 °C. The exposure temperatures were selected based on critical thermal events identified from prior Differential Scanning Calorimetry analysis, ensuring that the evaluation reflects service-relevant thermal conditions rather than arbitrary temperature selection [18].

All thermal exposure experiments were conducted for a fixed duration under an inert argon atmosphere to minimise oxidation and prevent environmental degradation during heating. The exposure duration was chosen to allow sufficient time for thermally activated diffusion and phase evolution to occur while avoiding excessive degradation that could obscure comparative stability trends. The overall Thermal Stability Analysis procedure integrates thermal analysis, controlled isothermal exposure, and post-exposure evaluation of microstructural and mechanical stability, enabling a systematic and stability-oriented assessment of material behaviour. The overall Thermal Stability Analysis (TSA) procedure adopted in this study is schematically illustrated in Figure 1.

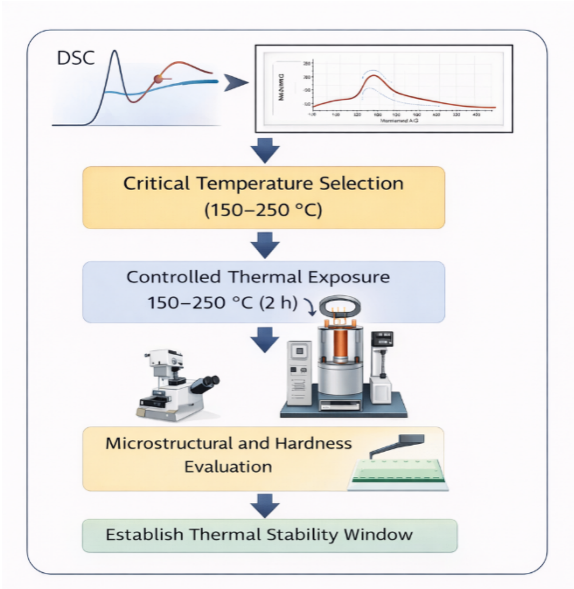

Fig. 1. Schematic of Thermal Stability Analysis (TSA) Framework

As shown in Figure 1, critical thermal exposure temperatures were first identified based on DSC results, followed by controlled thermal exposure and subsequent evaluation of microstructural stability and hardness retention. The TSA framework employed in this study integrates thermal analysis, controlled isothermal exposure, and post-exposure evaluation of microstructural and mechanical stability. This systematic approach enables a stability-oriented assessment of material behaviour under sustained thermal exposure and facilitates direct comparison between reinforced and unreinforced systems.

The thermal exposure conditions employed for the Thermal Stability Analysis are summarised in Table 1.

Table 1
Thermal exposure conditions for TSA

Sample	Exposure Temperature (°C)	Duration (h)	Atmosphere
AZ91	150	2	Argon
AZ91	175	2	Argon
AZ91-CNT	200	2	Argon
AZ91-CNT	225	2	Argon
AZ91-CNT	250	2	Argon

2.3 Microstructural Characterisation

Following thermal exposure, samples were prepared using standard metallographic techniques and examined using optical microscopy. Microstructural analysis focused on evaluating the morphology, continuity, and distribution of the β-$Mg_{17}Al_{12}$ intermetallic phase, as these characteristics are directly associated with the thermal stability and mechanical performance of AZ91-based alloys [19]. Particular attention was given to identifying signs of β-phase dissolution, coarsening, and redistribution as indicators of microstructural degradation during thermal exposure.

2.4 Hardness Measurement

Vickers microhardness testing was performed to assess the mechanical stability of the samples after thermal exposure. Multiple indentations were taken at different locations on each sample to minimise the influence of local microstructural heterogeneity and to ensure statistical reliability. The average hardness value was reported for each exposure condition, providing a quantitative measure of hardness retention and resistance to thermal softening [20].

3. Results

3.1 Hardness Evolution under Controlled Thermal Exposure

The hardness evolution of unreinforced AZ91 alloy and AZ91-CNT magnesium nanocomposites under controlled thermal exposure provides direct insight into their resistance to thermal softening. As illustrated in Figure 2, the unreinforced AZ91 alloy exhibits a progressive reduction in hardness with increasing exposure temperature, with significant degradation observed beyond 175 °C. This behaviour is attributed to the thermal instability of the β-$Mg_{17}Al_{12}$ intermetallic phase, which undergoes dissolution and coarsening at intermediate temperatures, thereby reducing its strengthening contribution [21,22].

In contrast, the AZ91–CNT nanocomposites demonstrate markedly improved hardness retention across the investigated temperature range. Hardness values remain relatively stable up to 200 °C, indicating enhanced resistance to thermal softening compared to the unreinforced alloy. The quantitative hardness values and corresponding retention percentages for both materials are summarised in Table 2, which clearly shows the superior hardness retention of the CNT-reinforced samples. The improved hardness stability is attributed to the presence of carbon nanotubes, which act as barriers to dislocation motion and suppress recovery processes during thermal exposure. In addition, CNTs restrict atomic diffusion within the magnesium matrix, thereby delaying phase instability and preserving mechanical strength under prolonged exposure [23].

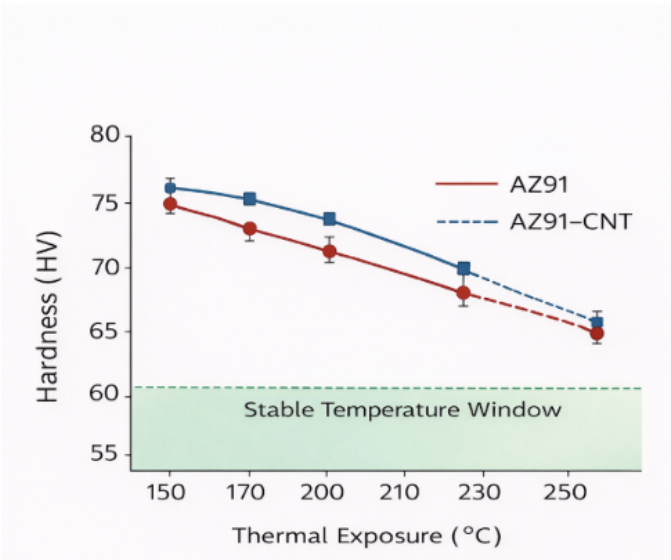

Fig. 2. Hardness retention vs thermal exposure temperature

The hardness values and corresponding retention percentages of AZ91 and AZ91-CNT samples after thermal exposure are summarised in Table 2.

Table 2
Hardness retention after thermal exposure

Sample	Temperature (°C)	Hardness (HV)	Retention (%)
AZ91	150	72	96
AZ91	200	61	81
AZ91-CNT	150	78	98
AZ91-CNT	200	74	93

3.2 Microstructural Stability and Phase Behaviour

Microstructural observations further support the hardness retention results and provide direct evidence of the stabilising effect of CNT reinforcement. Optical micrographs presented in Figure 3 show that the unreinforced AZ91 alloy exhibits pronounced coarsening and partial dissolution of the β-$Mg_{17}Al_{12}$ phase at relatively low exposure temperatures. Such microstructural degradation disrupts the continuity of strengthening phases along grain boundaries, contributing to the observed reduction in hardness [24].

In contrast, AZ91–CNT nanocomposites retain a finer and more continuous β-phase distribution following thermal exposure up to 200 °C, as shown in Figure 3. The preservation of β-phase morphology indicates that CNTs effectively delay phase coarsening and dissolution during thermal exposure. This stabilising behaviour is attributed to the role of CNTs as microstructural pinning sites, which restrict grain boundary migration and inhibit aluminium diffusion that drives β-phase instability [25]. Although microstructural degradation is observed at higher exposure temperatures for both materials, the onset and severity of degradation are significantly reduced in the CNT-reinforced samples.

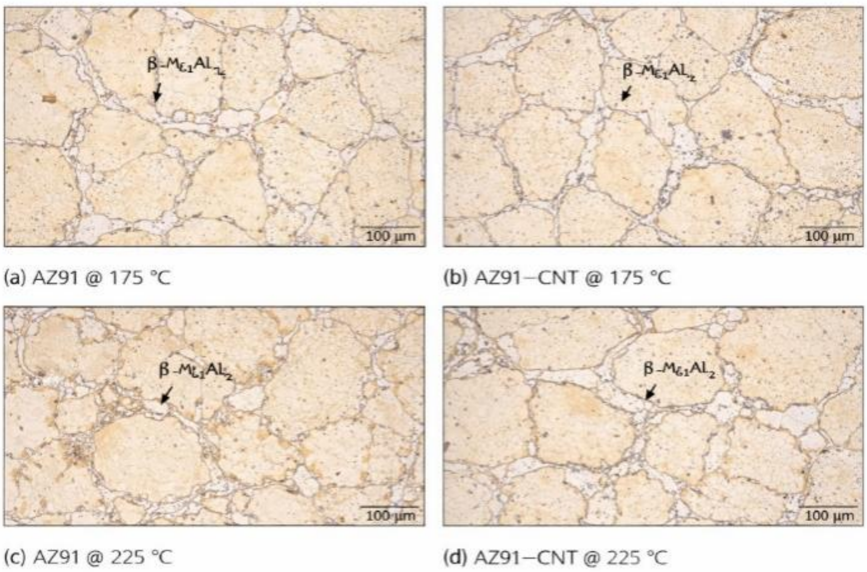

Fig. 3. Optical micrographs showing the microstructural stability

3.3 Thermal Stability Window Based on TSA

By correlating hardness retention data with microstructural observations, a TSA-based thermal stability window can be established for both materials. As summarised schematically in Figure 4 and quantitatively in Table 3, the unreinforced AZ91 alloy exhibits a stable operating temperature range limited to approximately 175 °C, beyond which rapid microstructural degradation and mechanical softening occur. In contrast, AZ91–CNT nanocomposites maintain stable microstructural and mechanical characteristics up to approximately 200 °C, representing a clear extension of the usable service temperature range [26].

The establishment of this stability window provides a more realistic assessment of thermal performance compared to conventional transformation temperatures obtained from Differential Scanning Calorimetry. Unlike DSC, which captures thermodynamic events under continuous heating, TSA accounts for time-dependent degradation mechanisms under sustained thermal exposure, enabling the identification of practical operating limits that are directly relevant to service conditions [27].

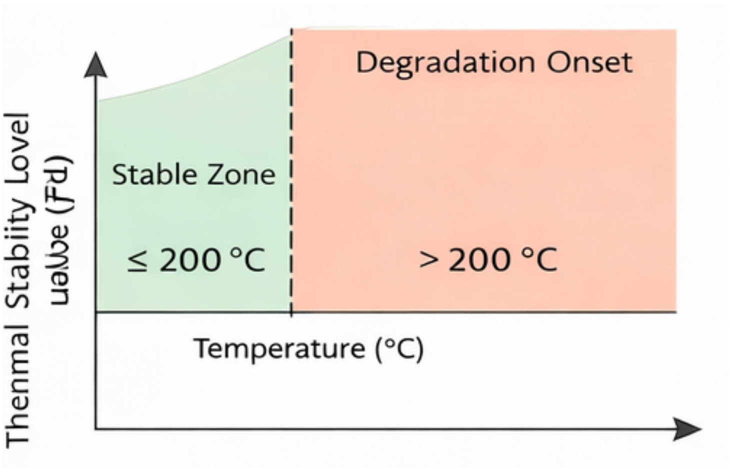

Fig. 4. Thermal stability window diagram

A summary of the thermal stability assessment for AZ91 and AZ91–CNT nanocomposites based on TSA results is presented in Table 3.

Table 3
Optical micrographs showing the microstructural stability

Material	Stable Temperature Range (°C)	Degradation Onset (°C)	Dominant Mechanism
AZ91	≤175	~180	β-phase dissolution
AZ91–CNT	≤200	~225	Delayed β-phase instability

3.4 Correlation between TSA and DSC Findings

The TSA results show strong agreement with DSC-identified critical thermal events, validating the integration of these two techniques. While DSC identifies the onset of phase transformations, TSA confirms whether these transformations translate into microstructural degradation and mechanical instability under isothermal exposure. The delayed degradation observed in AZ91–CNT nanocomposites indicates that CNT reinforcement effectively shifts the practical stability limit to higher temperatures, even when thermodynamic transformation thresholds remain unchanged [28].

This correlation highlights the importance of complementing DSC with TSA for comprehensive thermal evaluation. The combined approach bridges the gap between thermodynamic behaviour and service-relevant material performance, enabling more accurate prediction of long-term thermal stability in magnesium-based nanocomposites.

3.5 Limitations and Future Work

Although this study provides valuable insights into the thermal stability of AZ91-CNT nanocomposites, it is limited to short-term thermal exposure and hardness-based evaluation. Future work should include long-term creep testing, tensile evaluation after thermal exposure, and advanced characterisation techniques such as SEM and TEM.

4. Conclusions

This study has successfully conducted a comprehensive Thermal Stability Analysis of AZ91-CNT magnesium nanocomposites subjected to controlled thermal exposure, with the primary objective of evaluating their stability under service-relevant elevated temperature conditions. By integrating DSC-informed temperature selection with systematic isothermal thermal exposure, microstructural examination, and hardness evaluation, this work provides a stability-oriented assessment that extends beyond conventional thermal characterisation approaches.

The results clearly demonstrate that carbon nanotube reinforcement significantly enhances the thermal stability of AZ91 magnesium alloy. Compared with unreinforced AZ91, the AZ91-CNT nanocomposites exhibited superior hardness retention and delayed microstructural degradation during thermal exposure. In particular, the CNT-reinforced samples maintained stable mechanical and microstructural characteristics within the temperature range of 150-200°C, whereas the unreinforced alloy showed earlier onset of thermal softening and β-$Mg_{17}Al_{12}$ phase instability. The preservation of hardness and finer β-phase morphology confirms that CNTs act as effective diffusion barriers and microstructural pinning sites, restricting atomic mobility and suppressing premature phase dissolution and coarsening.

The TSA-based stability window established in this study reveals a clear extension of the practical operating temperature range for AZ91-CNT nanocomposites compared to the unreinforced alloy. Unlike transformation temperatures obtained solely from Differential Scanning Calorimetry, the TSA approach captures time-dependent degradation mechanisms and directly correlates thermal exposure conditions with microstructural and mechanical stability. The strong agreement between TSA results and DSC-identified thermal events further validates the reliability of this integrated evaluation framework.

Although this study provides valuable insight into the thermal stability enhancement of AZ91-CNT nanocomposites, it is limited to short-term thermal exposure and hardness-based mechanical evaluation. The current assessment primarily focuses on hardness retention and optical microstructural observations, which, while effective for identifying stability trends, do not fully capture long-term deformation behaviour or failure mechanisms under sustained thermal loading.

Future work should therefore include long-term creep testing and tensile evaluation following thermal exposure to provide a more comprehensive understanding of mechanical performance degradation at elevated temperatures. In addition, advanced characterisation techniques such as scanning electron microscopy and transmission electron microscopy should be employed to elucidate

CNT–matrix interfacial behaviour, nanoscale diffusion processes, and detailed phase evolution during thermal exposure. Such investigations would enable a deeper mechanistic understanding of thermal stabilisation in magnesium nanocomposites and support further optimisation of CNT-reinforced AZ91 alloys for demanding engineering applications.

Overall, this study establishes Thermal Stability Analysis as an effective and service-relevant methodology for evaluating the high-temperature performance of magnesium-based nanocomposites and highlights the potential of carbon nanotube reinforcement in overcoming the thermal limitations of conventional AZ91 magnesium alloy.

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References

[1] Mordike, B. L., and T. J. M. S. Ebert. "Magnesium: properties—applications—potential." *Materials Science and Engineering: A* 302, no. 1 (2001): 37-45. https://doi.org/10.1016/S0921-5093(00)01351-4
[2] Pollock, Tresa M. "Weight loss with magnesium alloys." *Science* 328, no. 5981 (2010): 986-987. https://doi.org/10.1126/science.1182846
[3] Friedrich, Horst E., and Barry L. Mordike. *Magnesium technology: metallurgy, design data, applications*. Berlin, Heidelberg: Springer Berlin Heidelberg, 2006.https://doi.org/10.1007/978-3-540-30812-1
[4] Luo, A. A. “Magnesium Casting Technology for Structural Applications.” Materials Science Forum 419–422 (2002): 57–66. https://doi.org/10.4028/www.scientific.net/MSF.419-422.57
[5] Kim, W. J., Y. G. Lee, and J. B. Lee. “Microstructure and Mechanical Properties of AZ91 Magnesium Alloy.” Materials Science and Engineering: A 499, no. 1–2 (2009): 213–218. https://doi.org/10.1016/j.msea.2008.09.061
[6] Zhou, W., and Z. M. Xu. “DSC Study of Phase Transformation in Magnesium Alloys.” Materials Characterization 38, no. 3 (1997): 187–194. https://doi.org/10.1016/S1044-5803(96)00108-5
[7] Singh, I. B., and V. C. Srivastava. “Differential Scanning Calorimetry Study of Phase Transformations in Magnesium Alloys.” Thermochimica Acta 617 (2015): 128–135. https://doi.org/10.1016/j.tca.2015.08.016
[8] Zhang, J., R. J. Perez, and M. Gupta. “Enhancement of Mechanical Properties of Magnesium Using Carbon Nanotubes.” Scripta Materialia 54, no. 2 (2006): 347–352. https://doi.org/10.1016/j.scriptamat.2005.09.014
[9] Hassan, S. F., and M. Gupta. “Development of Magnesium-Based Composites Using Carbon Nanotubes as Reinforcement.” Composites Science and Technology 66, no. 13 (2006): 2067–2074. https://doi.org/10.1016/j.compscitech.2005.10.017
[10] Rashad, M., F. Pan, A. Tang, M. Asif, S. Hussain, J. Gou, J. Mao, and H. Hu. “Effect of Carbon Nanotubes Addition on Mechanical and Thermal Properties of Magnesium Alloys.” Journal of Alloys and Compounds 603 (2014): 111–118. https://doi.org/10.1016/j.jallcom.2014.02.181
[11] Gupta, M., and S. M. L. Nai. Magnesium, Magnesium Alloys, and Magnesium Composites. Hoboken: Wiley, 2011. https://doi.org/10.1002/9780470958254
[12] Wang, X., K. Wu, K. Deng, and K. Nie. “Thermal Stability and Phase Evolution of Magnesium Alloys during Thermal Exposure.” Journal of Materials Science 47, no. 18 (2012): 6601–6609. https://doi.org/10.1007/s10853-012-6503-4
[13] Zhu, S. M., J. F. Nie, and A. J. Morton. “Microstructure and Thermal Stability of Magnesium Alloys at Elevated Temperatures.” Acta Materialia 59, no. 6 (2011): 2471–2483. https://doi.org/10.1016/j.actamat.2010.12.042
[14] Song, Guangling. "Recent progress in corrosion and protection of magnesium alloys." *Advanced engineering materials* 7, no. 7 (2005): 563-586. https://doi.org/10.1002/adem.200500013
[15] Aghion, E., B. Bronfin, and D. Eliezer. "The role of the magnesium industry in protecting the environment." *Journal of materials processing technology* 117, no. 3 (2001): 381-385. https://doi.org/10.1016/S0924-0136(01)00703-9
[16] Tjong, Sie Chin. "Recent progress in the development and properties of novel metal matrix nanocomposites reinforced with carbon nanotubes and graphene nanosheets." *Materials Science and Engineering: R: Reports* 74, no. 10 (2013): 281-350. https://doi.org/10.1016/j.mser.2013.08.001
[17] Surappa, Mirle Krishnegowda. "Aluminium matrix composites: Challenges and opportunities." *Sadhana* 28, no. 1 (2003): 319-334.https://doi.org/10.1007/BF02717141
[18] Clyne, Trevor William, and Philip John Withers. *An introduction to metal matrix composites*. Cambridge university press, 1993. https://doi.org/10.1017/CBO9781139170830

[19] Rashad, M., F. Pan, and A. Tang. "Carbon Nanotube Reinforced Magnesium Composites: A Review." Journal of Magnesium and Alloys 2, no. 4 (2014): 353–368. https://doi.org/10.1016/j.jma.2014.11.001
[20] Singh, A., and M. Gupta. "Enhancing the High-Temperature Performance of Magnesium Alloys through Nanoreinforcements." Materials & Design 92 (2016): 869–879. https://doi.org/10.1016/j.matdes.2015.12.084