

STUDY THE RESIDUAL STRESSES IN COMPOSITES MATERIALS USING X-RAY TECHNIQUE

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ABSTRACT

Residual stresses significantly influence the properties and performance of epoxy-based coatings, with their origins rooted in various factors encountered during production and application. The residual stresses within the laminated carbon steel metal alloy/epoxy composites were investigated. We employ X-ray diffraction to measure these residual stresses. In this paper, the hardness, density, and residual stresses were studied using X-ray techniques for coating at different curing temperatures (25, 50, and 75 °C) as coating materials deposited on carbon steel alloy substrates using the spin coating technique. The highest hardness was recorded at 50 °C, indicating optimal curing conditions and balanced network formation. The density tests show that curing temperature impacts the microstructure of the epoxy. The highest density was observed at 25 °C, indicating a more compact polymer matrix. The results demonstrated that the effect of curing temperature on the epoxy coatings cured at low temperatures exhibited low residual stress values compared to the coatings cured at higher temperatures (75 °C).

Keywords: curing temperature, residual stress, X-ray method, carbon steel.

NOMENCLATURE

$\sigma_{res.}$	Residual stresses
ϵ	Elastic modulus
$d\psi$	The distance between planes perpendicular to the surface
dn	The distance between planes perpendicular to the surface.
ν	Poisson's ratio
ψ	The sample's rotational angles
ρ	Density
m	Mass
V	Volume

Received: June 17, 2025.

Accepted: September 01, 2025.

INTRODUCTION

Residual stresses are the stress fields that remain after a mechanical process has deformed a material, even when no external loads are applied. One example is thermal strain, which occurs when heating or cooling is not uniform. Discontinuity in deformation as a result of temperature change is caused by mismatched thermal expansion coefficients, and incompatible deformation is created by plastic deformation. The component's history of processing and the material characteristics that link mechanical process to deformation behaviour are the two primary determinants of residual stress (Cheng, and Finnie, 2007). Residual strains can be created during manufacturing and/or use by operations like mechanical forming, heat treatment, or welding. Heat treatment, machining, secondary processing, and assembly are some of the macroscopic sources of residual stresses. On a microscopic level, they are typically caused by differences in thermal expansion coefficients, yield stresses, rigidities, or phase shifts (such cure shrinking) among various components. Any given material or component could have both types of stress simultaneously (Colpo, F, 2006). Epoxy coatings' residual stress evolution is a relatively unexplored field of study. Polymeric coatings are widely used in engineering applications to enhance the surface properties of substrates by providing corrosion protection, thermal and electrical insulation, and improved mechanical integrity. However, one of the major challenges affecting the durability and performance of such coatings is the development of residual stresses during the manufacturing process, particularly throughout the curing and cooling stages.

Residual stresses in polymeric coatings can develop from various factors, including volumetric shrinkage of the polymer matrix during curing, thermal mismatch between the coating and the substrate, and uneven temperature gradients. These stresses can build up either within the coating layer or at the interface, potentially causing cracking, delamination, or early failure of the coated system (Wang, et al. 2024).

The magnitude and distribution of residual stresses are influenced by various parameters such as curing temperature and time, coating thickness, substrate properties, and the presence of nano-fillers, which can alter the thermal and mechanical behavior of the coating. Understanding these stresses is critical for optimizing coating performance and ensuring long-term structural reliability. To assess and predict residual stresses, researchers have adopted several advanced techniques, including X-ray diffraction (XRD) particularly the $\sin^2\psi$ method for experimental stress measurements. These approaches provide valuable insights into stress mechanisms and help in tailoring coating systems for specific operational conditions. Assessed the bi-material interface's stainless steel-epoxy surface for thermal residual stresses using X-ray techniques. He demonstrates that X-ray technique can be used to identify surface stress state changes caused by thermal residual stresses (Kanerva, M. et al. 2012). Used X-ray diffraction to measure the residual tension in polyamide sheet polymer. Experimental measurements were taken using a diffractometer that operates through a gearbox approach. They went over some of the issues with detecting polymer coating residual stresses and demonstrated that the X-ray methodology works well for this purpose (Taisei, et al. 2015).

Investigated the residual stresses developed in epoxy-steel laminates as a result of thermal curing shrinkage, post-cure cooling, and hygroscopic expansion due to moisture absorption. The results indicated that the initial curing shrinkage-induced residual stresses were relatively small, while substantial residual stresses were generated during post-curing cooling due to thermal contraction mismatches between epoxy and steel (Baran et al. 2015).

Used $\sin^2\psi$ based on X-ray diffraction (XRD) approach to investigate the residual stresses essentially found in wear protection coatings. Specifically, the coating of wear was conducted via 95% Al_2O_3 and 5wt.% SiC substrate besides using AlNi alloy as bond coat that being generated via flame spraying method. This in turn has confirmed compressive residual stresses of (-325.67 MPa) for the recent coating. It is noteworthy to mention that (-325.67 MPa) of compression residual stress might be enhanced via the employment of a novel mixture of Al_2O_3 and SiC substrate (Dai, J. et al. 2019).

The present study aims to investigate the development and distribution of residual stresses in epoxy-based polymeric coatings subjected to different curing temperatures, applied on metallic substrates (A36 carbon steel) used for crude oil storage tanks is achieved.

MATERIALS AND METHODS

Experimental Part

Materials

The coatings in question are composed of two liquid components, the base and the hardener, which are mixed before being applied to the alloy surface. One of these components is DGEBA, which stands for bisphenol-A-(epichlorhydrin) epoxy resin. The mix volume ratio of the base and hardener in this particular Sikadur 52 epoxy-based coating is 1:2. This coating is manufactured by Sika Yapi Kimyasallari A.S. and is used for the plain coating of tanks without external additives. The study used properties of a carbon steel A36 substrate with a purity level of 99.0%. The substrate was prepared for deposition process using emery papers in different grades (3000, 1000, 300, 180, 120, 100, 80, 60, 40) in order to smoothing or roughing the substrates surface before the deposition process using polishing machine MoPao series (160E). After polishing process the surfaces of substrates washed and cleaned by Trichloroethane as chemical cleaner to remove the residual impurities and fats to be prepared for coating process

Laminated Samples Preparation

The ASTM A36 carbon steel substrate, with dimensions (30 * 30 mm and a thickness of 6 mm) was cut and machined from plates to the desired shape. The ASTM-E0915-96R02 (ASTM, 2010) standard is used to determine the specimen dimensions. The carbon steel alloy substrate (A36) was used to apply the coating material using the spin coating technique. The laminated composite samples in this work were prepared using the spin coating technique by depositing the epoxy at different temperatures (25 °C, 50 °C, and 75 °C). Figure 1 displays specimens both before and after coatings, along with a scanning

electron microscope (SEM) image of the cross-section that is 400X magnified to illustrate the thickness of the epoxy coatings. Carbon steel substrates were coated with Sikadur by spin casting at 236 -245.3 μm film thickness (Xu, D. et al. 202), which is considered typical. The coating thickness was confirmed using scanning electron microscope equipment.

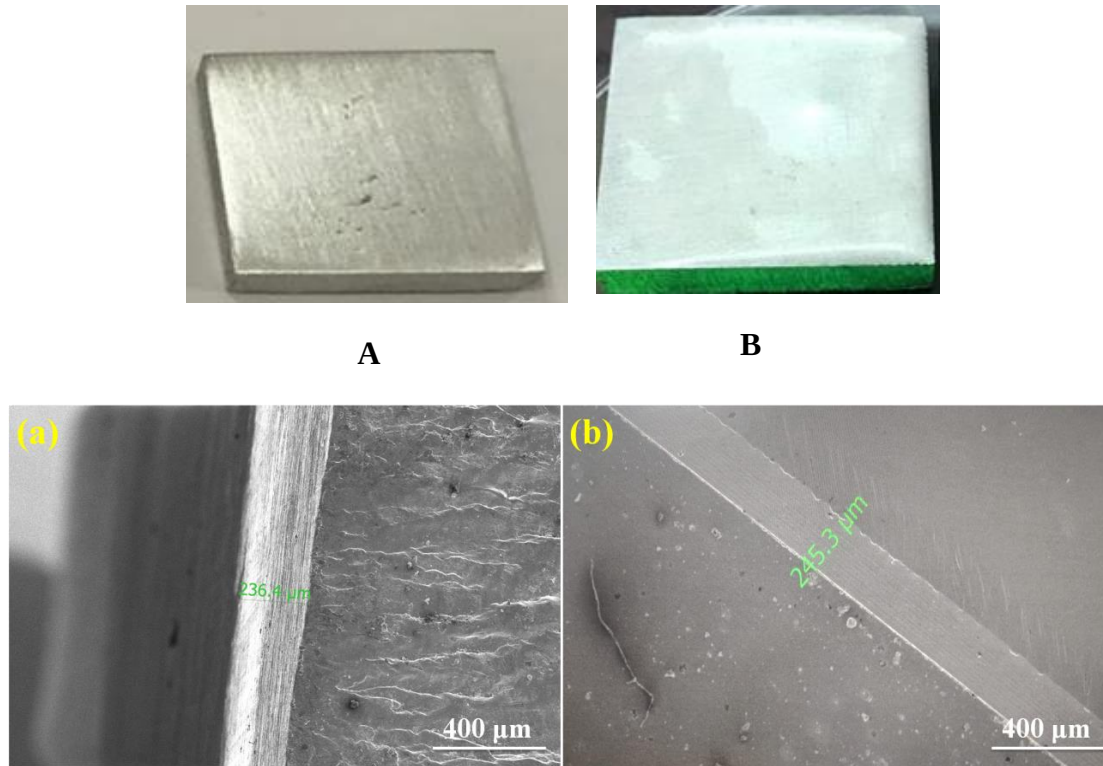


Fig. 1. Specimen before coating (A), after coating (B), SEM of sample surface after coating (a and b).

Coating samples were left to cure at three different temperatures (25, 50, and 75°C) in order to evaluate how the curing temperature affected the residual strains caused by solidification. According to the data sheets provided by the manufacturers of different epoxy-based coatings, the curing period can reach 6 hours (Mucha, M., et al. 2020). For the X-ray measurement, three specimens were utilized, with the $\sin^2 \psi$ technique being employed.

Residual stress measured by X-ray

The strains in the coatings crystal lattice were measured along the tangential direction using X-ray diffraction ($\sin^2 \psi$ method). Assuming a biaxial state of stresses, the associated stress is computed from the elastic constants (modulus of elasticity and Poisson's ratio) (Anderoglu, O. 2005) see Table 1.

Table 1. Epoxy coating (model A) elastic modulus and Poisson's ratio obtained from tensile test.

Cure temperature °C	Elastic modulus (MPa)	Poisson's ratio
25	3800	0.302
50	4400	0.313
75	5500	0.319

When measuring strain using X-ray diffraction, it is assumed that the epoxy-based coating's crystal lattice plane exhibits a homogeneous linear elastic distortion. The diffractometer was used to measure the d-spacing for various ψ values. The slope for each sample was determined using the d-spacing vs. $\sin^2\psi$ curve. The residual stress was then estimated using the following relationship (Gazzara, C. P. 1983) as shown in Table 2:

$$\sigma_{res.} = \frac{E}{(1 + \nu) \sin^2 \psi} \frac{d_{\psi} - d_n}{d_n} \quad (1)$$

where d_{ψ} represents the distance between planes perpendicular to the surface (in micrometres) and d_n represents the distance between planes perpendicular to the surface (in micrometres) E stands for the coatings' elastic modulus (GPa), ν for Poisson's ratio, and ψ for the sample's rotational angles.

Table 2. Representation of Magnitude of Residual Stress (MPa)

Sample	Residual Stress (MPa)
Fe alloy + Ep at 25 °C	-33.15
Fe alloy + Ep at 50 °C	-18.76
Fe alloy + Ep at 75 °C	-56.10

The intricate structure of the epoxy coating, which comprises lamellae, pores, micro-cracks, and more, makes it difficult to quantify mechanical parameters such as elastic modulus and Poisson's ratio. To assess the elastic modulus and Poisson's ratio of different types of epoxy coatings, tensile tests are conducted on dog bone tensile test specimens cut according to ASTM D-638 type V, as illustrated in Figure 2. Until failure happens, the load is gradually increased as deformation (stress with strain) progresses. A Microcomputer Controlled Electronic Universal Testing Machine, housing the tensile testing apparatus, may be found in the Engineering Materials College of the University of Babylon. Additional glass plates that resemble the ASTM D638-type V tensile test specimen in terms of size and shape are also utilised (Maxwell, and Turnbull, 2003; Nuutinen, 2019). The Sikadur epoxy coatings were cast into the tensile specimens using the moulds. The glass

plates were secured using rubber binding. Temperatures of 25, 50, and 75°C were used to cure the specimens. Tensile tests are conducted on the specimens as if they were elastic materials. For the purpose of tensile testing, three specimens were prepared.

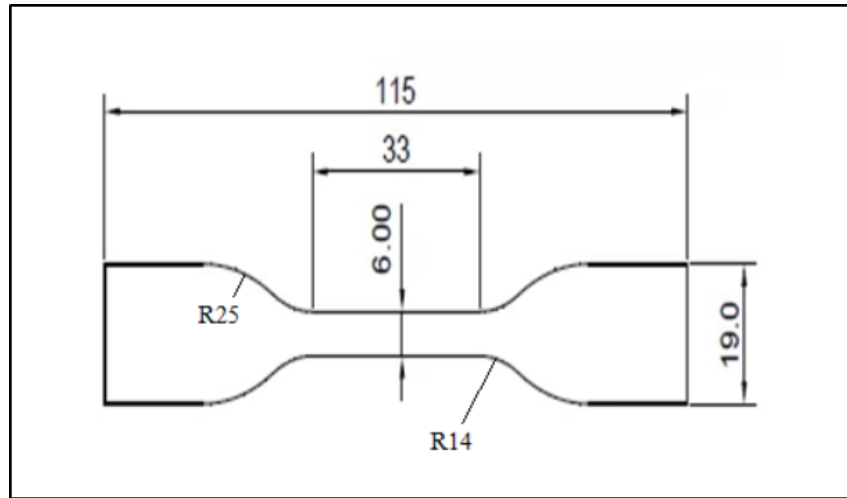


Fig. 2. Tensile test specimen.

DENSITY TEST

Density is defined as mass per unit volume, measured in kilograms per cubic meter (kg/m³). In polymeric materials like epoxy, several factors influence the density during the curing process, including

- Temperature for curing.
- Rate of reaction and crosslinking of polymers.
- Reduction in volume during the polymerization process.
- Presence of microvoids or air bubbles.

The density of all prepared samples calculated using the traditional method through the density law equation (Maurer, R. J., et al. 2019).

$$\rho = \frac{m}{V} \quad (2)$$

Where ρ is the density (g/cm³), m is the mass (g) and V is the sample volume (cm³). The samples were cut into cubes with dimensions of (1*1*1 cm) and the mass was weighted using a balance (Balance four degrees) and the volume calculated of each sample cube, the density was calculated by dividing the mass to the volume.

RESULTS AND DISCUSSION

Energy-dispersive X-ray spectroscopy (EDS)

An elemental examination of the specimen was conducted using Energy Dispersive X-ray Spectroscopy (EDS) to determine the surface chemical composition of the alloy. The investigation revealed the presence of the following components: Iron (Fe), Carbon (C), all of which are consistent with the standard composition of carbon steel A36 Alloy, along with small amounts of Fluorine (F), Calcium (Ca), and Potassium (K). The outcomes were introduced in two structures: weight percentage (Wt%) and atomic percentage (At%), as shown in Table 3. Iron and carbon were the predominant components in the Fe alloy. Figures 3 show an SEM image of carbon steel A36, along with its elemental distribution. The EDS range (Figure 4) shows the trademark peaks of each component, corresponding to the photon energies during electronic transitions. The examination provided evidence that the example was made of carbon steel A36, as the discovery of the elements and their composition and weight rates were consistent with the standard composition of the alloy.

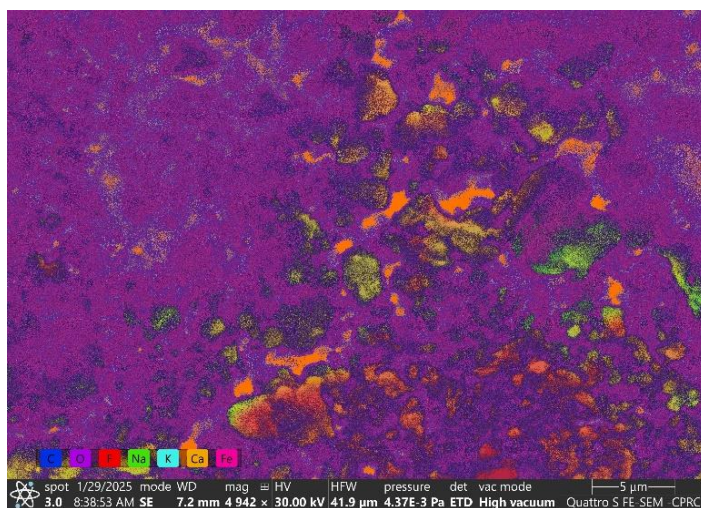


Fig. 3. SEM image of carbon steel alloy A36.

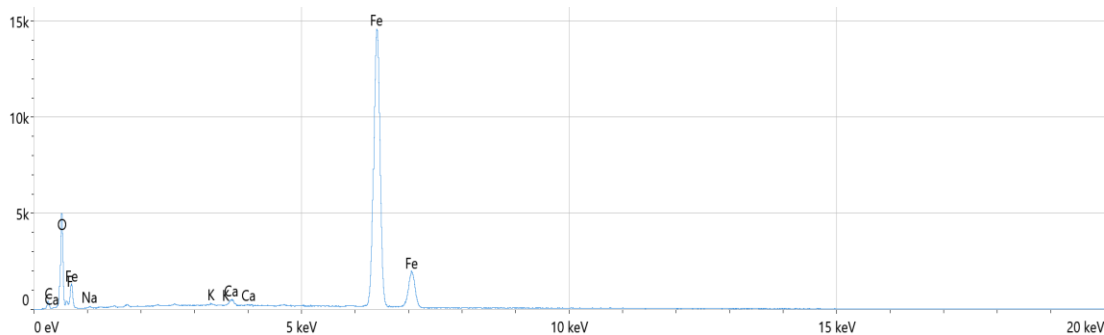


Fig. 4. Energy dispersive spectroscopy of ready Fe A36 alloy.

Table 3: Chemical composition of Fe A36 alloy.

Element	Atomic %	Atomic % Error	Weight %	Weight % Error
C	9.0	0.1	3.4	0.1
O	8.9	0.2	4.5	0.1
F	0.0	---	0.0	---
Na	1.1	0.0	0.8	0.0
K	0.1	0.0	0.1	0.0
Ca	0.5	0.0	0.7	0.0
Fe	80.3	0.0	90.5	0.0

DENSITY TEST

At 25°C (density = 1.08 kg/m³), this is room temperature, where the curing reaction is relatively slow. Slow curing allows for better molecular arrangement and fewer voids. A more compact and uniform polymer network can form, leading to higher density (Ellis, B. (Ed.). 1993). At 50°C (density = 1.04 kg/m³), higher temperature accelerates the curing process. However, rapid reaction may trap air bubbles or volatile compounds before they can escape. This leads to the formation of micro-voids, which reduces the overall density (Lee, and Neville, 1967). At 75°C (density = 1.06 kg/m³), the curing is faster and more complete. The higher temperature may help to remove trapped gases and allow better crosslinking. As a result, density increases again, approaching the value at 25°C (Pascault, and Williams, (Eds.). 2009) as shown in Figure 5. At 50°C resulted in the most balanced combination of properties, including reduced residual stress and acceptable mechanical performance.

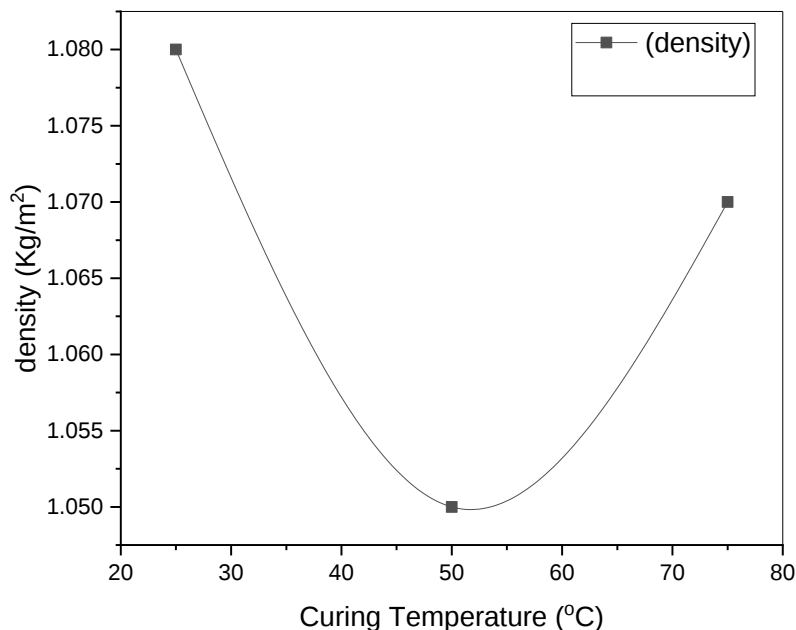


Fig. 5. Density test specimen.

HARDNESS TEST

A Shore D durometer instrument (TIME 5431 model, China), at Babylon university/ College of Materials Engineering, was used to test cylindrical specimens with at least 3 mm thickness, following ASTM (D2240) standards (Saeed and Z. K. M. Al-Obad 2023). The curing temperature significantly affects the mechanical properties of epoxy resins, such as hardness. The extent of crosslinking achieved during curing is vital for the final mechanical performance. Typically, increasing the curing temperature speeds up the crosslinking reaction, resulting in a denser and more rigid polymer structure.

At 25°C Moderate Hardness, This figure reflects a partially cured network with insufficient crosslinking. At room temperature, the movement of molecular chains is limited, and the curing reaction proceeds slowly, leading to a loosely bonded polymer structure (Yu, Y., et.al 2006) as shown in Figure 6. At 50°C maximum hardness achieved, this temperature seems ideal for the curing process, enabling adequate molecular mobility and reaction rate. The result is a more uniform and tightly crosslinked network, leading to increased hardness (Carbas, et.al 2014). At 75 °C Slight reduction in hardness, although higher temperatures promote additional curing, various factors can lead to a decrease in hardness. Rapid curing may create excessive internal stresses. Additionally, over-curing can induce brittleness and possible microcracking. There is also a risk of uneven crosslinking or partial thermal degradation (Pascault, and Williams, (Eds.). 2009). The experimental results obtained in this study are consistent with several published works and

demonstrate that 50°C is the optimal curing temperature to achieve the highest hardness. This is due to the enhanced crosslinking reactions at that temperature, leading to a denser polymer network.

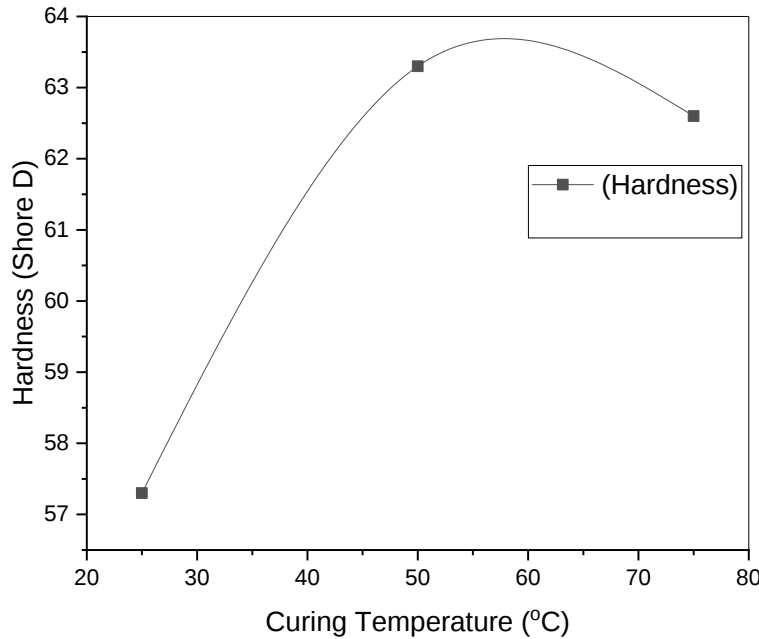


Fig. 6. Hardness test.

RESIDUAL STRESSES CALCULATED USING X-RAY DIFFRACTION

For the internal stress examination, internal stresses were assessed using X-ray diffraction ($\sin^2 \psi$) for all prepared samples, including pure epoxy composites. The stress examination is one of the critical tests that determine the mechanical behavior of laminated materials. All prepared samples were analyzed.

Table 2 presents the residual stress values obtained through X-ray diffraction using the ($\sin^2 \psi$) method at different temperatures. The residual stress readings for the A36 carbon steel alloy coated with a 0.236 mm layer of epoxy, measured at various temperatures (25°C, 50°C, and 75°C), were -33.15 MPa, -18.76 MPa, and -56.10 MPa, respectively as illustrated in Figure 7. Coefficient of Thermal Expansion (CTE) Mismatch, The greater the curing temperature, the larger the potential thermal contraction mismatch upon cooling. Also, it can be noted that all residual stresses are compressive stresses. As seen, the compressive residual stress would be produced because the reaction between the base and hardener during mixing to produce the epoxy coating completely occurs after the coating is applied. Also, the residual stress in the epoxy coating is compressive due to the smaller coefficient of thermal expansion of the epoxy coat than that of the substrate of steel coating. There is a significant increase in residual stress values with an increase in curing

temperatures of coatings, whereas these values are lower at lower temperatures. It is found from Figure 7 that the best curing temperature 50°C, which provides a lower value of residual stress (Yu, Y., et.al 2006). At 25°C curing: The low temperature during curing results in limited polymer chain movement. As it cools, the epoxy shrinks more than the steel substrate, causing high compressive residual stresses (Francis, et.al 2002). At 50°C curing: A higher curing temperature enhances internal molecular movement and allows for some stress relaxation during cooling, resulting in lower compressive residual stress (Rastak, et.al 2021). However, when metal were coated with epoxy and cured at 75°C, the residual stresses at the interface between the coating and the metal substrate were higher in steel. This happens due to the larger mismatch in the coefficient of thermal expansion (CTE) between epoxy and steel, which leads to more significant thermal contraction differences during cooling. Additionally, steel's higher Young's modulus makes it more resistant to deformation, resulting in greater stress buildup (Li, Q., et.al 2022).

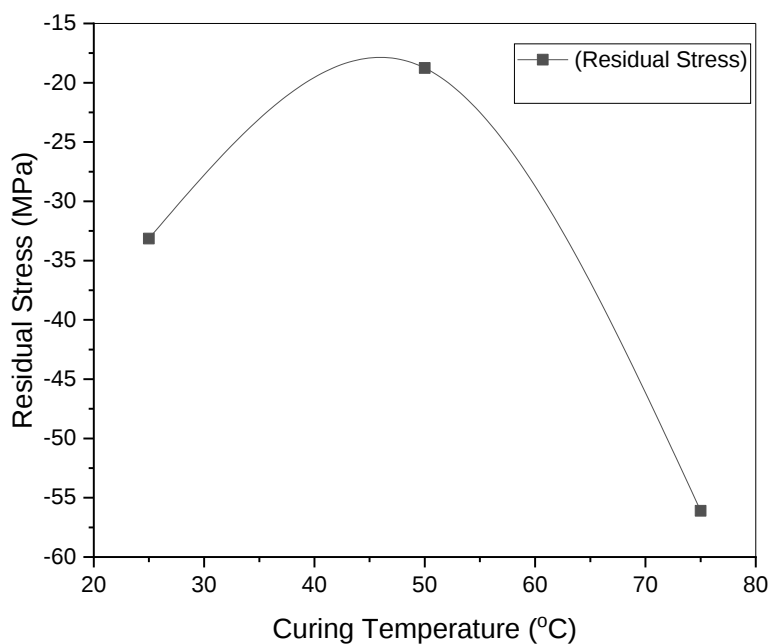


Fig. 7. Variation of residual stresses with curing temperature at 25, 50, and 75°C.

CONCLUSION

1. In this research, we looked at how different curing conditions affect the levels of residual stress. For instance, the curing temperature has become an important consideration; at lower temperatures, residual thermal stresses are lower than at higher temperatures.
2. The results show that the residual stresses determined by X-ray diffraction are -33.15 MPa at 25°C, -18.76 MPa at 50°C, and -56.10 MPa at 75°C.

3. It has been determined that all residual stresses are compressive.
4. The XRD technique, using the ($\sin^2\psi$) method, proved to be very effective in accurately measuring surface residual stresses, especially when focusing on the epoxy coating layer by using a low grazing incidence angle to reduce substrate influence.
5. The lowest hardness occurred at 25°C, while the highest was at 50°C.
6. The density test demonstrates that the curing temperature affects the microstructure of epoxy. The highest density was observed at 25°C, indicating a more compact polymer matrix. The dip at 50°C suggests void formation, while the increase at 75°C signifies better curing efficiency. This type of analysis provides indirect insight into the internal quality of the cured epoxy.

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