Two-Phase Intercritical Annealing AR400 Steel: Effects on Phase Dispersion and Impact Resistance for Mining Applications

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Abstract

Low-carbon AR400 abrasion-resistant steels are utilised as wear packages prolonging the production life of ground-engaging tools on earth-moving machines in South Africa's mining sector. While AR400 steel is renowned for its tribological properties and wear resistance, components frequently fail prematurely due to inadequate impact absorption, necessitating premature removal from the heavy-duty production environment. This study investigates a two-phase intercritical annealing process to enhance the ductility and impact energy absorption of AR400 steel. Phase 1 involves heat treating the specimens at 1000°C for 25 minutes, then oil quenching. Phase 2 reheats the specimens to 750°C for 20 minutes, followed by cool water quenching. The efficacy of this treatment was evaluated by comparing the mechanical properties, phase dispersions, and general grain morphology of the heat-treated specimens with those of the untreated control specimens. Microstructural analysis using optical and scanning electron microscopy revealed a transformation from the martensitic phase dispersion in the control specimens, typically associated with AR400, to a primarily bainitic microstructure in the intercritically annealed samples. This change improved the impact energy absorption and ductility of the 20 mm specimen by 200%. However, the results were inconsistent across material thickness. The 12 mm and 16 mm specimens showed a decrease in impact resistivity and approximately 40% reduction in material hardness and strength, irrespective of material thickness, compromising wear resistance. The results demonstrate that while the proposed two-phase intercritical annealing heat treatment can enhance impact properties, the concurrent reduction in hardness limits its practical application in industries requiring wear and impact resistance.

Keywords: Intercritical Annealing, Brinell Hardness, Phase Dispersion, Oil-Quenching.

1. Introduction

Abrasion-resistant (AR) steels are used in high-wear and impact applications as liner plates in wear packages for earth-moving machines, extending the production life of ground-engaging tools operating in platinum mining, while protecting the structural frame of the component beneath, these wear packages are periodically replaced once the integrity of the liner plates has been compromised [1]. AR steels are specifically used in wear and impact environments for the material's renowned tribological properties, combined with increased toughness and impact resistance, making them ideal for applications requiring long-term resistance to wear commonly associated with Heavy-Duty mining operations in South Africa [2]. However, a three-year research study on the production cycle of the wear packages revealed that only 54% of the wear packages met the minimum production cycle requirements, and the failure mechanism is often attributed to impacts sustained during operation before the maximum wear life of the liner plates was reached, significantly increasing component downtime, and resulting in a loss of valuable operational efficiency [3]. The Minerals Council South

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Africa found that the mining industry employs approximately 477,000 people and is one of South Africa's most significant contributors to Gross Domestic Product (GDP) [4]. In contrast, the Platinum Group Metals (PGM) mining sector accounted for the largest share of income for the mining industry, at 41.2% in 2022 [5], warranting the necessity of ground-engaging tools and earthmoving machines to be repaired or manufactured to a standard that will ensure the components maintain their minimum production life cycle, maximizing efficiency, and production outputs.

As such, there is a need for application-specific steels with superior wear resistance and tribology, combined with improved impact absorption, for heavy-duty wear and impact applications in the open-cast mining industry. However, it is a well-established principle that material toughness and hardness are inversely proportional; a harder, more rigid material might have improved tribological properties, but, in turn, is proportionately brittle, with reduced impact energy absorption capabilities [6]. One of the challenges encountered when identifying suitable materials for liner plates is that AR steels are known to be categorised or grouped into a class based solely on the nominal hardness of the material, often established through the verification of surface hardness alone, and not on the mechanical properties of the steel [7], with no regulatory standard governing the wear performance [8], and have no specific standard for the mechanical properties or chemical spectrometry [9]. Although the mechanical properties of AR steels depend primarily on the spectrometry and alloying element concentration, the microstructure, phase dispersion, grain size, morphology, distribution, and dislocations significantly influence the behaviour and application of the materials. These microstructural characteristics are a consequence of manufacturing conditions, such as the heat-treatment temperature and quenching rates during quenching processes [10], which depend on the manufacturer and their specific heat-treatment capabilities and protocols.

Research shows that the impact resistivity of dual-phase steels, used in the automotive industry, has been improved through intercritical annealing without compromising the materials' hardness [11]. This material processing has not yet been applied to abrasion-resistant steels, and as such, this study investigates a novel two-phase intercritical annealing process. The results and methodology detailed in this article are representative of a broader study investigating intercritical annealing of abrasion-resistant materials under various processing conditions, specifically varying hold times and quenching media during phase one intercritical annealing.

The objectives of this investigation were to characterise the general phase dispersion and grain morphology while qualifying the subsequent changes in the mechanical properties, specifically the material hardness and impact energy absorption, and finally to conclude on the efficacy of the proposed methodology and heat treatment processing for materials used in the South African mining industry.

While a previous investigation and publication detail the results of variations in hold time and quenching media, this article focuses specifically on analysing the efficacy of a two-phase heat treatment at 1000°C for 25 minutes, followed by oil quenching, concentrating on the microstructural characterisation to quantify the phase dispersion and morphology, and a comprehensive comparison of the mechanical properties relevant to mining applications.

2. Literature Review

Since grain morphology, phase dispersion, and associated mechanical properties are fundamentally dependent on heat-treatment processing and cooling rates, this section reviews relevant literature on the relationships of heat-treatment parameters, intercritical annealing mechanisms, and their influence on microstructural development.

Vogel emphasises that, ideally, quenching must occur immediately after removing products from the furnace, and the quenching rate should be high enough to achieve maximum mechanical properties, usually exceeding the critical quenching rate for the specific alloy [12]. Goncalves found that the toughness and mechanical properties of materials tend to decrease with slow cooling rates due to carbide precipitation at grain boundaries [13], which aligns with the ferritic and pearlitic dispersions observed in slow-cooled materials. However, as shown in Figure 1, the transformation of austenite during heat treatment and, ultimately, the phase dispersions that influence mechanical properties are also affected by the cooling rate. Rapid cooling from the critical temperature typically results in a primarily martensitic dispersion, associated with high hardness. At the same time, moderately cooled, oil-quenched specimens develop a bainitic dispersion featuring both upper and lower bainite, and slowly cooled specimens tend to form pearlitic and ferritic dispersions with significantly lower hardness [14].

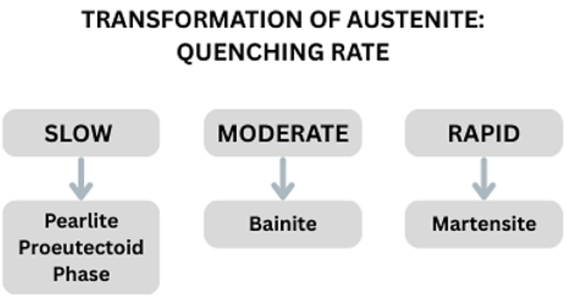

Figure 1. Transformation of Austenite Cooling Rate [15].

2.1. Heat treatment processing

Heat treatment is used to introduce toughness, ductility, improved mechanical properties, and hardness to a material, and can also reduce internal stresses; it also refines the microstructure, depending on its purpose [16]. AR steels typically have a combined martensitic and bainitic dispersion, with approximately 60–80% martensite [17]. However, bainitic dispersions, formed during moderate cooling, have been found to improve the material toughness, proportionately increasing material ductility and impact energy absorption [18]. The resulting toughness caused by the presence of bainite, a combination of essentially bainitic ferrite and cementite, is formed at high temperatures when the carbon partition of supersaturated ferrite is retained in the form of austenite, and carbides precipitate in coarse cementite [19]. Rapidly cooled specimens do not have sufficient time to transform [20], as the carbon atoms that dissolved in the austenite to carry out diffusion movements and form cementite are not present, resulting in the formation of martensite [16].

2.2. Intercritical annealing

Intercritical annealing, as a heat-treatment mechanism, can be used to generate fixed ferrite and austenite volume fractions that precipitate as controlled proportions of ferrite, martensite, and bainite, refining the microstructure [21]. The optimal strength-to-ductility ratio and grain morphology are generated by intercritical annealing and are frequently used to refine grain morphology and microstructures [22]. Intercritical annealing is achieved by heat-treating a material approximately 30°C above Ac1, the temperature at which austenite begins to form, typically 700 –750°C for low-carbon alloy steels [23]. However, Singh notes that the ideal temperature range for intercritical annealing is between the upper and lower transformation temperatures [24]. Cao found that steels that have been intercritically annealed at 800°C exhibited improved tensile ductility associated with the long-term continued transformation-induced plasticity (TRIP) effect of austenite [25], while Ayatollahi ascertained that optimal results for intercritical annealing are obtained when the material is heated between the Ac1 and Ac3 temperature range at 750°C for 10 minutes and water quenched, as illustrated in Figure 2 [26]. Alfirano states that low-carbon alloy steels develop a combined ferritic and martensitic dispersion when rapidly quenched after 6-18 minutes between the transformation temperatures [27].

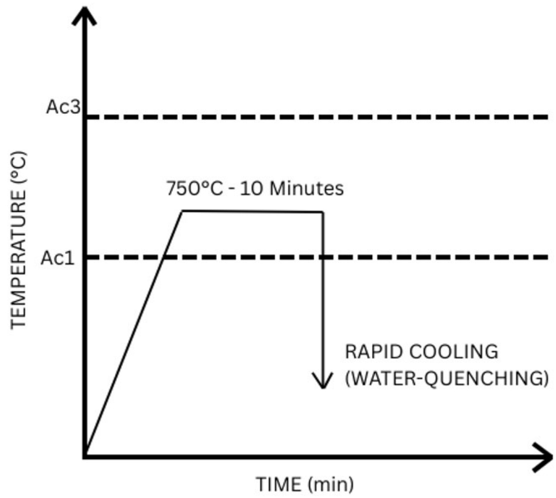

Figure 2. Intercritical Annealing Processing Time-Temperature Graph [14].

2.3. Quenching media

Research shows that optimised combined strength-ductility in low-carbon alloy steel is achieved when non-isothermal partitioning processing and energy-efficient quenching are used [28], and it depends on the selected quenching medium and cooling rate. Rapid quenching, typically associated with martensite formation, is ideal for increasing material hardness and wear resistance, but it also tends to make the material more brittle [13]. This prompts consideration of medium- to rapid-quenching media to assist with grain refinement and the formation of desired phase dispersions.

2.3.1. Oil quenching

Quenching oil 32 is an industry-grade standard-speed quenching medium, similar to low-viscosity mineral oil, ideal for maintaining a consistent quenching medium temperature during cooling, and with a high flashpoint, reducing the risk of combustion [29]. The oxidation stability of a synthetic quenching oil prevents acid build-up and staining for a superior surface finish while reducing the risk of distortion or cracking during the quenching process [29]. Food-grade vegetable oils have been shown to increase the impact energy when used as a quenching oil, thereby improving material toughness, impact strength, hardness, yield strength, and elongation, whereas mineral or transmission oils are ideal for applications requiring a rapid quench rate but are not biodegradable [30].

2.3.2. Water quenching

Water-quenching is generally used as the preferred quenching medium in the production of abrasion-resistant steels [31], and can be used as an effective medium to rapidly cool specimens following heat treatment at critical temperatures, depending on the water temperature; however, unlike oil-quenched media, the severity of the cooling can lead to distortion and cracks in some materials [30].

However, the rapid cooling forms martensite, which is responsible for the material's increased hardness, tribological properties, and subsequent wear resistance. Especially in the large-scale production of abrasion-resistant wear plates, water quenching is considered a low-cost quenching alternative; notably faster than oil and air cooling, water quenching introduces a significant amount of martensite a typically hard and brittle structure known to improve material hardness and tribology but subsequently also make the material more brittle, and stiff and reduces the impact strength and impact energy absorption of the material [32].

The grain size and martensitic lath width decrease with increasing cooling rate; rapidly quenched steel typically exhibits fine, evenly dispersed grain morphologies [33].

2.3.3. Air Quenching

Similarly, the air-cooled alternative, like water-quenching, is a low-cost quenching method; however, it takes significantly longer and can be used to reduce distortion in sufficiently hardenable materials [34]. However, slower air-cooling results in pearlitic and ferritic dispersions, associated with materials that have reduced hardness but increased impact energy absorption and ductility [14].

Air is rarely used as a quenching or cooling medium because it is a gas, which results in inconsistent outcomes due to the inability to create a controlled environment, and it is significantly more time-consuming than oil- and water-based quenching alternatives [16].

2.4. Typical AR400 material properties

Although the metallurgical properties of the AR400 control specimens will be determined through metallurgical testing, the recommended nominal values for spectrometry, hardness, and mechanical properties of low-carbon alloy steel were selected, as no governing standard specifies minimum or maximum values for properties other than hardness.

AR400 is associated with a nominal material hardness of 400 HB and the carbon equivalent and chemical composition detailed in Tables 1 and 2, respectively [35], the recommended Brinell Hardness range for this respective material is between 370 HB and 430 HB according to ISO specifications [36], however, it is dependent mainly on manufacturer specifications and surface hardness testing [1].

Table 1. Carbon Equivalent for AR400 **[35]**.

Plate Thickness	CEV	CET
10 mm	0.45	0.30
25 mm	0.49	0.32
40 mm	0.56	0.32

Table 2. Typical Alloying AR400 [35].

Element	Concentration (w%)	Element	Concentration (w%)
C	0.25	Ni	0.7
Si	0.7	Mo	0.5
Mn	1.7	B	0.005
P	0.025	Cu	0.3
S	0.015	Nb	0.05
Cr	1.5		

The carbon equivalent, chemical properties, and mechanical properties detailed in Table 3 are dependent on the manufacturer's specifications and heat-treatment processes and are not governed by a specific regulatory standard [37]. They are used to determine the material's hardenability and the likelihood of crack propagation in welded steel [38]. AR400 is typically water-cooled, achieving high material hardness through rapid cooling from the austenitizing temperature, resulting in a martensitic microstructure. However, once the material has been formed or hot-formed, its hardness can only be maintained by secondary heat treatment at 250°C [35].

Table 3. Typical Mechanical Properties of AR400 [34].

Material Property	Nominal Range
Hardness (BHN)	360 – 440
Yield Strength (MPa)	800 - 1069
Tensile Strength (MPa)	1241
Elongation	12 – 14%

The literature review establishes a need to improve the mechanical properties of abrasion-resistant steels for application-specific operation in the South African mining industry. The variation in material property standardisation and governing standards for mechanical and chemical properties, in conjunction with the lack of application-specific heat-treatment process guidelines, provides a foundation for investigating the efficacy of intercritical annealing as a heat-treatment mechanism to address the premature failure of abrasion-resistant plates in combined high-wear and impact environments.

3. Materials and Experimental Framework

The study first establishes the existing trend for the spectrometry, surface hardness, mechanical properties, and impact energy absorption capabilities of the AR400 control specimens, not only for comparison to the recommended standard found in the literature review and to verify the veracity of the material certificates but also as a baseline to compare against the metallurgical testing results of the same properties for the intercritically annealed specimens as a point of departure.

The experimentation framework illustrated in Figure 3 outlines the research approach and methodology used in this study.

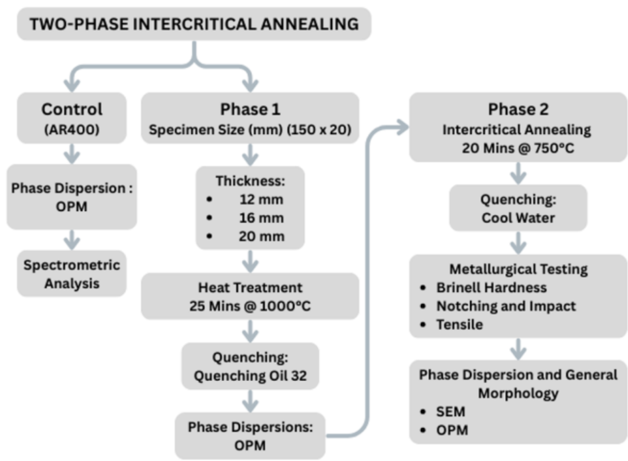

Figure 3. Experimental Framework.

The study focused on determining the effects of the two-phase intercritical annealing process across three material thicknesses commonly used as wear package specifications: 12 mm, 16 mm, and 20 mm thick AR400 steel.

In Phase 1, the AR400 abrasion-resistant steel specimens were heat-treated in a Labcon L-1200 slab element furnace at 1000°C for 25 minutes, followed by oil 32 quenching to offset the brittleness caused by martensite in the AR400 steel phase dispersions. The aim was to increase the impact energy absorption by increasing the proportion of bainitic structures and dispersions.

An OPM analysis is used to determine the phase dispersions in the control specimens and the dispersion following the Phase 1 heat treatment. During Phase 2, the materials are further heat-treated at 750°C for 20 minutes, followed by water quenching. The general grain morphology and final phase dispersions following the completion of Phase 2 are established through an SEM analysis and compared to the phase dispersions of the Control specimens and the Phase 1 OPM analysis. The

JEOL JSM 6010 PLUS/LA Analytical Scanning Electron Microscope was used for the analysis of all SEM samples and micrographs.

The metallurgical testing was conducted according to the relevant International Organization for Standardization (ISO) specifications for abrasion-resistant steels, detailed in Table 4, which are often used and recommended as quality measures in South African mining applications and Quality Control Processes (QCP). The Tensile Testing was conducted using a SANS 600 kN (S/N 30704014) Universal Testing Machine (UTM), and all Impact testing was evaluated using a SANS KV2 Charpy impact testing machine (S/N 21210014).

Table 4. Mechanical Testing Specifications [1].

Mechanical Testing	ISO Specification
Brinell Hardness	ISO6506-1
Notching and Impact	ISO148-1
Tensile Test	ISO6892-1
Spectrometric Analysis	ISO6507-1
Micro Examination	OPM and SEM

The selection of the 25-minute hold time and quenching media was based on a preliminary review of intercritical annealing heat-treatment parameters. The focused analysis of a single quenching medium and hold time specifically supports the possibility of improving both material hardness and impact resistance in abrasion-resistant materials intended for industrial applications in the South African mining sector, where the premature failure of wear packages on ground equipment and earthmoving machines remains a significant operational challenge. The suggested experimental framework has not been previously investigated as a heat treatment process for AR400 liner plates and wear packages.

To ensure objectivity and avoid potential bias in microstructural interpretation, all phase dispersion, microstructure identification, and bainite quantification were independently verified by a certified metallurgical engineer with expertise in steel microstructure analysis.

3.1. Sample preparation

The as-received AR400 specimens were wire-cut into 150 mm x 20 mm segments for each material thickness, for both the two-phase intercritical annealing processing and the control analysis. For microstructural OPM and SEM analysis, the specimens were cut to size, ground, polished, and mounted for optical OPM and SEM analysis to determine the phase dispersion and general grain morphology of the intercritically annealed specimens. The specimens were etched in 2% Nital to reveal the microstructure and phase dispersion, and thoroughly cleaned and dried prior to the examination. The specimens for metallurgical testing were prepared in accordance with the relevant ISO specifications for the test.

3.2. Quenching Oil 32 Key Properties

Quenching oil is specifically used in applications where heat treatment is intended to harden carbon and low-alloy steels used in industry. It has lower oxidation rates and volatility, resulting in reduced consumption and safer operational environments. The specific key properties are listed in Table 5 below.

Table 5. Quenching Oil 32 Key Properties **[39].**

Property	Value
Density (20°C)	0.864 kg/L
Viscosity (40°C)	32 cSt
Viscosity (100°C)	5 cSt
Viscosity Index	100
Fire Point	248°C
Pour Point	-9°C
Flash Point C.O.C.	220°C
Neutralisation NO	0.001 Mg/(KOH)/g

4. Results and Discussion

This section evaluates and discusses the outcomes of the metallurgical and microanalytical analyses of the control and intercritically annealed specimens. The results of the metallurgical analysis conducted on the intercritically annealed specimens were compared to the properties of the control specimens established below to evaluate the effects of the proposed two-phase intercritical annealing on the material properties, hardness, phase dispersion, and whether the processing improved the impact resistivity of the material while maintaining the tribological properties.

Although the two-phase intercritical annealing is expected to affect the material hardness to a certain degree since the literature review reiterates the well-established principle that hardness and toughness are inversely proportional, if the processing is successful, it will not be affected to such an extent that the wear rate should be significantly altered.

4.1. Material properties - AR400 control specimen metallurgical testing

The results of the control specimens' material properties and metallurgical analysis were used as a basis for comparison to evaluate the efficacy of the proposed two-phase intercritical annealing process and the subsequent changes to the heat-treated specimens' material properties, as well as to determine whether the properties of the control specimens aligned with those found in the literature review.

4.1.1. Spectrometric analysis

The chemical analysis summarised in Table 6 confirmed that the spectrometry of the control specimens and the concentration of the alloying elements coincide with the typical standard for AR400 materials specified in the literature review and with the specifications listed on the accompanying material certificates within the allowable margin of error according to the South African National Accreditation System (SANAS).

However, the major, minor, and trace alloying elements are illustrated in Figures 4 to 6, respectively, showing that the Carbon wt% for the 12 mm specimen was 15% lower than that of the 16 mm and 20 mm control specimens. The diminished carbon content is typically associated with increased hardness due to extended hold time above the material's Ac3 temperature. Similar discrepancies in the concentration of alloying content were found with the concentration of Manganese (Mn), responsible for material toughness, increasing proportionately to the material thickness, while elements like Phosphorus (P), Sulfur (S), Chromium (Cr), and Molybdenum (Mo), amongst other seemingly inversely proportional to the material thickness, highlighting the lack of regulatory manufacturing standards for the alloying content in AR steels.

Table 6. Spectrometric analysis of AR400 Element wt% for 12mm, 16mm, and 20mm Control Specimens [1].

Element	12 mm	16 mm	20 mm
C	0.13	0.15	0.15
Si	0.35	0.34	0.36
Mn	0.87	0.96	1.21
P	0.017	0.014	0.013
S	0.007	0.005	0.004
Cr	0.33	0.38	0.35
Mo	0.17	0.16	0.01
Ni	0.01	0.01	0.01
Cu	0.03	0.03	0.02
Al	0.052	0.051	0.101
V	0.004	0.004	0.006
Ti	0.006	0.008	0.007
No	0.017	0.017	0.011
Co	0.01	0.012	0.006
W	0.038	0.037	0.034
Pb	0.009	0.01	0.008
B	0.0008	0.0007	0.0007
As	0.007	0.008	0.003
Sn	0.004	0.006	0.001
Sb	0.006	0.007	0.004
Fe	Rem	Rem	Rem

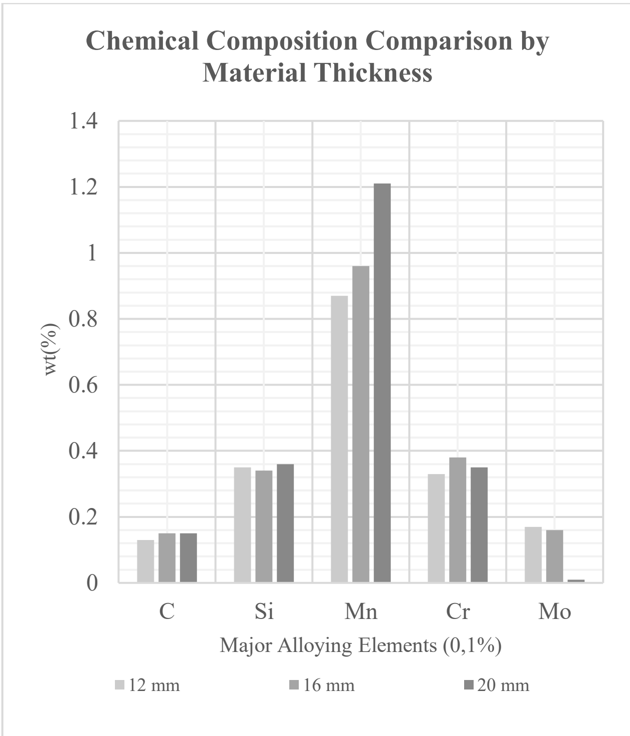

Figure 4. Chemical Composition Comparison by Material Thickness: Major Alloying Elements (> 0.1%).

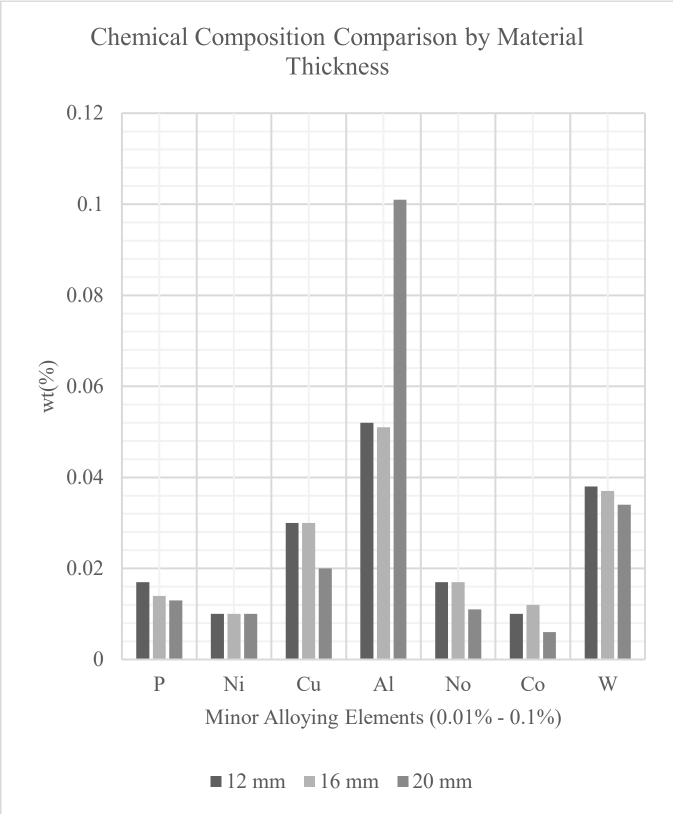

Figure 5. Chemical Composition Comparison by Material Thickness: Minor Alloying Elements (0.01% - 0.1%).

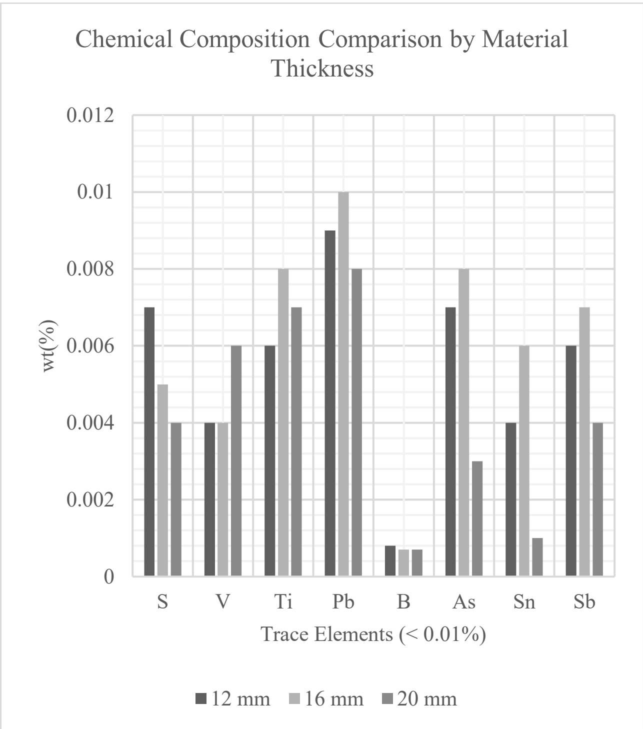

Figure 6. Chemical Composition Comparison by Material Thickness: Trace Elements (< 0.01%).

4.1.2. Mechanical testing

The metallurgical evaluation of the control specimens' mechanical properties, detailed in Tables 7 for the 12 mm, 16 mm, and 20 mm specimens, respectively, shows that the specimens' Ultimate Tensile Strength (UTS) comply with the typical properties associated with AR 400 steel and detailed on the material certificates within the allowable SANAS margin of error. However, the ISO specification for Brinell hardness testing of AR400 steel indicates a recommended hardness range of 370-430 Brinell, although the nominal hardness of AR400 is typically around 400 HB. A higher material hardness can increase brittleness and reduce impact energy absorption.

According to ISO 6506-1, Brinell hardness testing indicated that the evaluated material thicknesses did not fall within the nominal hardness range. The Brinell hardness for both the 16 mm and 20 mm specimens was approximately 3% above the specified range; however, it remained within the allowable SANAS margin of error.

The hardness of the 12 mm specimen was 12 % above the nominal range, which also accounted for the diminished carbon content observed in the spectrometric analyses. The metallurgical analyses showed that the materials' UTS, Yield stress, and elongation are consistent with the ISO specification and the minimum expected values for low-carbon alloy abrasion-resistant steels.

Table 7. Control Specimens – Mechanical Properties [1].

Property	12 mm	16 mm	20 mm
Brinell Hardness	477 HB	444 HB	444 HB
Yield Load	89.9 kN	143.2 kN	152.4 kN
Max Load	102.4 kN	164.0 kN	172.4 kN
Yield Stress	1173 MPa	1188 MPa	1230 MPa
UTS	1336 MPa	1360 MPa	1392 MPa
Elongation	11%	12%	12%

However, both the 12 mm and 16 mm specimens failed to meet the minimum average impact energy specification and were tested well below the recommended 45 Joules, as summarised in Table 8. Impact and notch testing were conducted at -40°C in accordance with the ISO specification. This is consistent with the increased hardness of the respective samples and their potential for greater material brittleness.

Table 8. Control Specimens – Impact Energy Absorbed [1].

	12 mm	16 mm	20 mm
Specimen Ave. Energy Absorbed (Joules KV(2))	39	29	56

4.1.3. Phase dispersion analysis

An OPM analysis, using optical microscopy, was used to evaluate the phase dispersions of the as-received control specimen and revealed that the AR400 specimens had a combined bainitic and martensitic phase dispersion, illustrated in Figures 7 to 9, consistent with the properties found in the literature review as the martensitic dispersion is typically associated with the high hardness of the material and is common for materials that have been rapidly quenched or water quenched after the initial heat treatment phase.

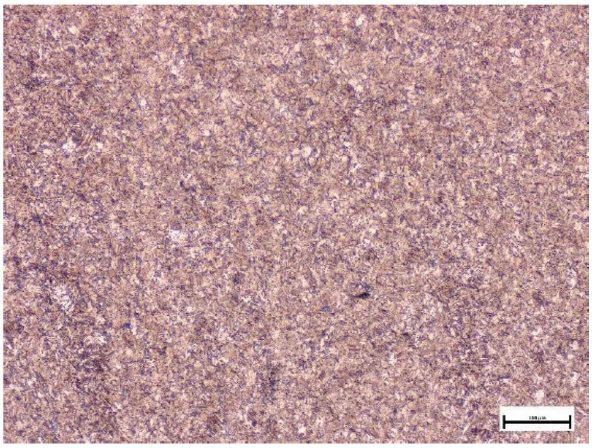

Figure 7. Control Specimens Phase Dispersion: 12mm etched with 2% Nital, Microstructure showing tempered martensite and bainite. x100 mag (100 µm scale bar).

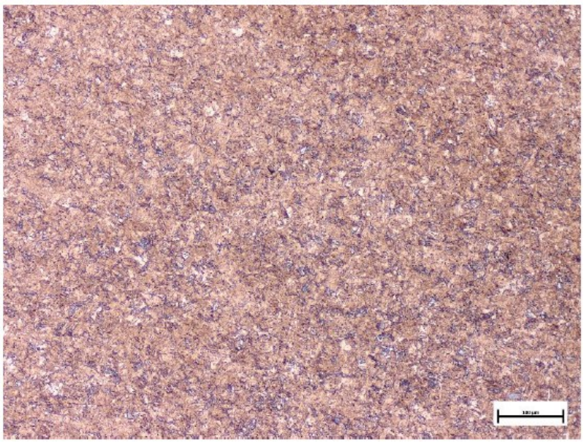

Figure 8. Control Specimens Phase Dispersion: 16 mm, etched with 2% Nital, Microstructure showing tempered martensite and bainite. x100 mag (100 µm scale bar).

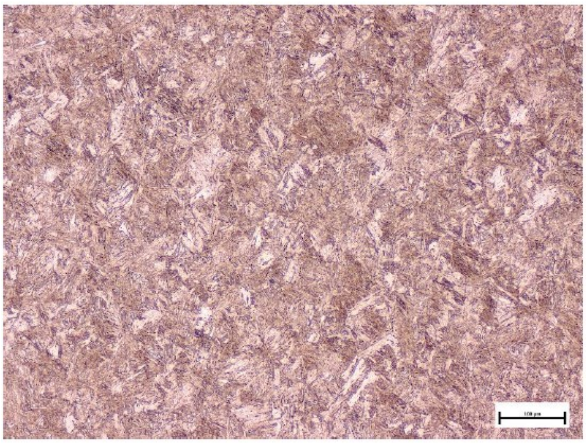

Figure 9. Control Specimens Phase Dispersion: 20 mm, etched with 2% Nital, Microstructure showing tempered martensite and bainite. x100 mag (100 µm scale bar).

4.2. Material properties - intercritically annealed specimen metallurgical testing

The results, evaluating the intercritically annealed specimens' material properties, are used to compare the established control specimens' parameters and determine the effects of the proposed heat-treatment mechanism on the materials' mechanical and tribological properties and impact energy-absorption capabilities.

4.2.1. Phase 1 – OPM phase dispersion analysis

The OPM analysis evaluates the phase dispersions of the specimens after 25 minutes of heat treatment at 1000°C, followed by oil quenching, as specified in the Phase 1 methodology.

The images in Figures 10 to 12 show that the materials' phase dispersion is consistent with that observed for materials cooled at a moderate rate following heat treatment at a critical temperature, with the inclusion of upper and lower bainitic dispersions due to oil quenching.

The OPM analysis revealed that the 20 mm specimen has a fully lower bainitic dispersion, whereas the presence of lower bainite decreases proportionately with increasing material thickness. The 16 mm specimen has an even distribution between upper and lower bainite, while the 12 mm specimen has a 35% and 65% upper and lower bainitic dispersion, respectively.

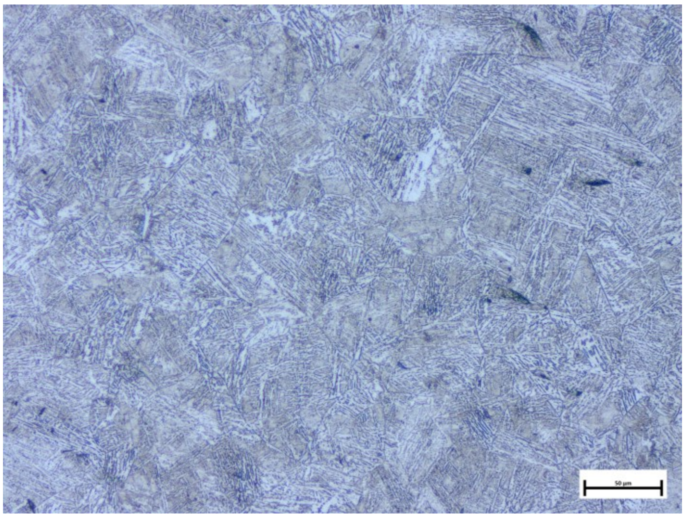

Figure 10. Phase 1 OPM Micro 12 mm – (50µm scale bar), etched in Nital-2.

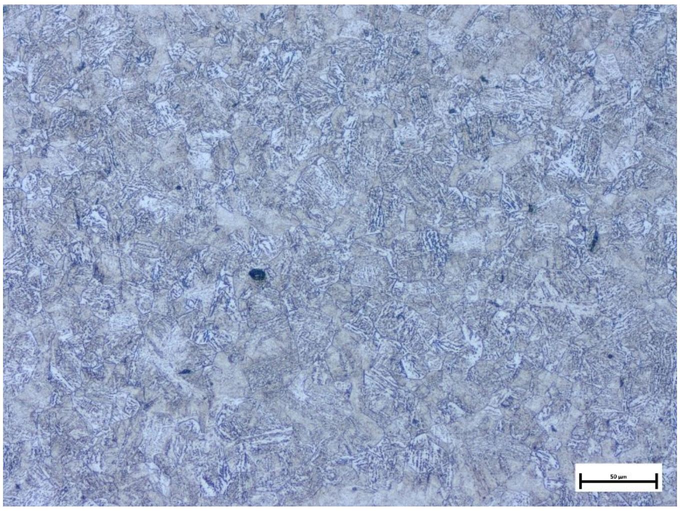

Figure 11. Phase 1 OPM Micro 16 mm - (50µm scale bar), etched in Nital-2.

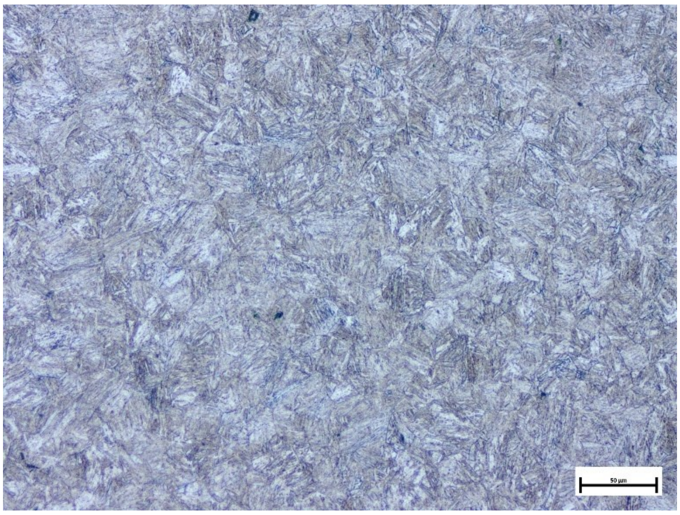

Figure 82. Phase 1 OPM Micro 20 mm - (50µm scale bar), etched in Nital-2

4.2.2. Analysis of mechanical properties

The metallurgical testing and sample preparation for the material parameters summarised in Table 9 below were conducted in accordance with the relevant ISO specifications. Compared with the same-thickness control specimens, the 12 mm and 16 mm specimens show no improvement in impact energy absorbed, but rather a significant decrease below the 45 Joule minimum average typically associated with low-carbon abrasion-resistant steels. This is despite the nearly 40% reduction in material hardness observed in the intercritical annealed specimens compared to the as-received specimens.

Table 9. Intercritically Annealed Specimens – Mechanical Properties.

Property	12 mm	16 mm	20 mm
Brinell Hardness	286 HB	298 HB	256 HB
Yield Load	17.20 kN	9.59 kN	15.2 kN
Max Load	21.29 kN	10.52 kN	19.57 kN
Yield Stress	623 MPa	759 MPa	549 MPa
UTS	795 MPa	829 MPa	711 MPa
Elongation	14%	17%	23%

As impact resistivity, summarised in Table 10, and material hardness are typically inversely proportional, an increase in the specimens' impact energy absorption is expected due to the reduced material hardness. All Charpy impact tests were conducted with n=3 specimens per condition at -40°C.

Table 10. Intercritically Annealed Specimens – Impact Energy Absorbed [1].

Energy Absorbed (Joules KV(2))	12 mm	16 mm	20 mm
Specimen 1	7	12	245
Specimen 2	8	15	240
Specimen 3	6	12	130
Specimen Ave.	7	13	205

In contrast to the pattern seen in the 12 mm and 16 mm specimens, the 20 mm specimen exhibited significantly increased impact energy absorption, nearly five times that of the minimum specification average and approximately four times that found in the control specimen. A one-way ANOVA confirms that thickness effects are statistically significant ($p < 0.001$), as the comparison with control specimens shows that the impact resistivity of the 12 mm specimen decreased by 82% and the 16 mm specimen decreased by 55%; however, the impact resistivity of the 20 mm specimen increased by 266%.

This could be attributed to the already-established superior impact energy absorption observed in the control specimen analysis and to the reduction in material hardness due to intercritical annealing. Similarly, the reduction in material hardness was reflected in the yield stress and UTS of the intercritically annealed specimens, which decreased by approximately 43%. As such, the inverse relationship between hardness and impact resistance was evident: thinner specimens (Table 9) retained higher hardness and exhibited lower impact energy, whereas the 20 mm specimens achieved lower hardness (256 HB) with substantially improved impact resistance. This variation could potentially be attributed to faster cooling rates in thinner sections, which prevent complete bainitic transformation.

The increase in elongation measured for each specimen was directly proportional to the material thickness, with the 20 mm specimens showing an 80% increase as a result of the proposed material processing and the 12 mm and 16 mm specimens showing a 30% and 42% increase, respectively

4.2.3. Phase 2 – General morphology and phase dispersion analysis

Following the completion of Phase 2 heat treatment, an OPM analysis of the intercritically annealed specimens, illustrated in Figures 13 to 15, revealed a primarily bainitic dispersion; however, as bainite is essentially a combination of cementite and pearlite, it can be challenging to distinguish optically as a final measure.

This necessitates SEM analysis to establish the general grain morphology and, ultimately, the effects of the proposed heat-treatment process.

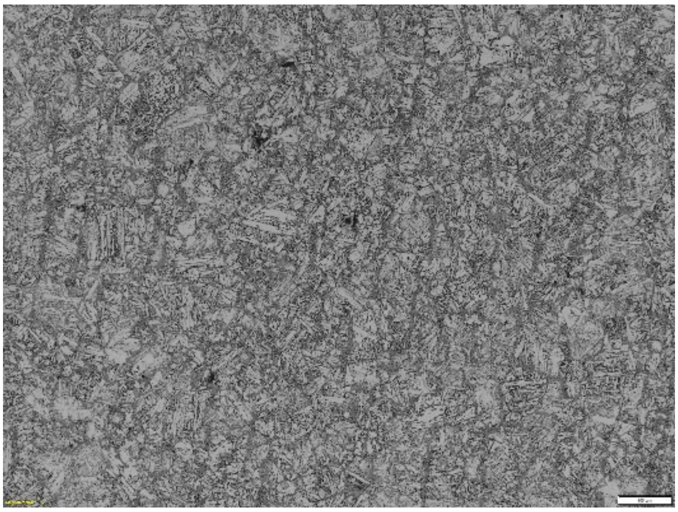

Figure 13. Intercritically Annealed Specimen Phase Dispersion:12mm, etched with 2% Nital, (200µm scale bar).

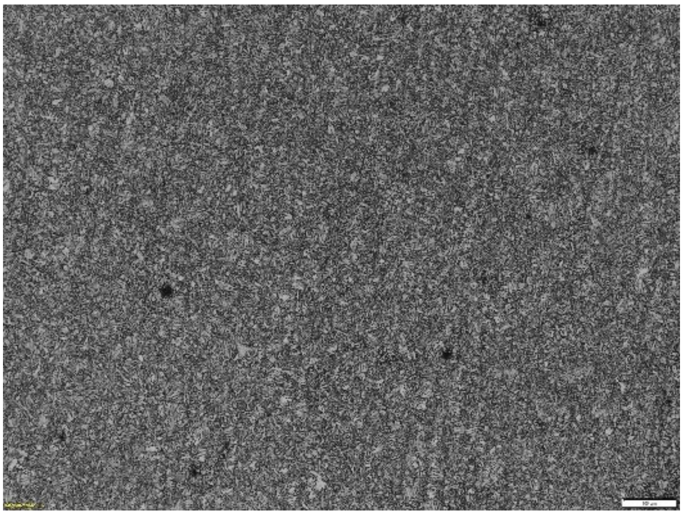

Figure 14. Intercritically Annealed Specimen Phase Dispersion:16mm, etched with 2% Nital, (200µm scale bar).

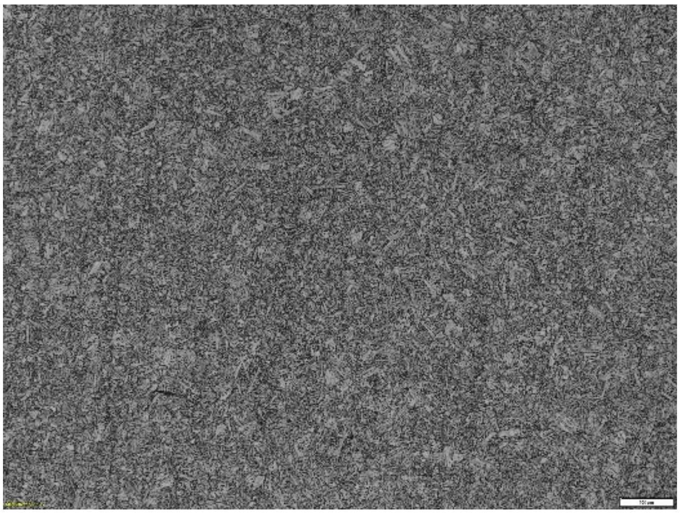

Figure 15. Intercritically Annealed Specimen Phase Dispersion: 20mm, etched with 2% Nital, (200µm scale bar).

The purpose of the SEM analysis, illustrated in Figures 16 to 18 for the 12 mm, 16 mm, and 20 mm specimens, respectively, is not to conduct an in-depth analysis of the specimen microstructure but to verify the phase dispersions found in Phase 1 and Phase 2 OMP analysis and to ascertain the general grain morphology and structure.

The results of the SEM analysis confirm that the intercritically annealed specimens have a primarily bainitic dispersion and that the percentage increase of lower bainite is directly proportional to the increase in material thickness. However, closer inspection shows that the 12 mm specimen has a 40% upper and 60% lower bainitic dispersion, while the 16 mm specimens exhibit a 20% and 80% upper and lower bainite dispersion, and the 20 mm specimen has a 10% and 90% upper and lower bainite dispersion, respectively.

The grains of the intercritically annealed specimens are coarse with a polygonal equiaxed grain morphology and the inclusion of some needle-like structures consistent with moderately cooled low-carbon alloy steels. A finer grain structure has been known to increase the material's impact resistance by reducing stress concentrations at grain boundaries.

The discrepancies in microstructure and phase dispersion observed between the OPM and SEM results, especially in the thinner material samples, can be attributed to differences in imaging techniques and magnification levels. The OPM is used for an expedited, cost-effective overall assessment of the phase distribution at the phase 1 process hold point, as it is at a lower level of magnification. At the same time, the SEM provides significantly higher-resolution micrographs that enable more accurate evaluation of grain morphology and quantification of bainite.

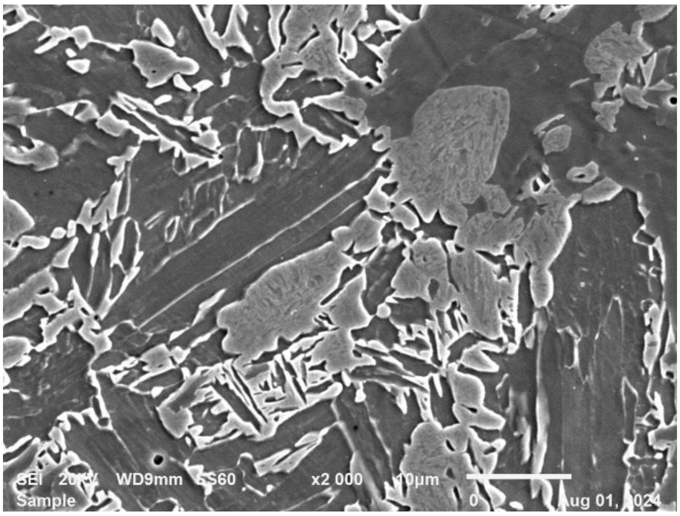

Figure 16. SEM 12 mm specimen x2000, (10μm scale bar), etched in Nital-2, Intercritically Annealed.

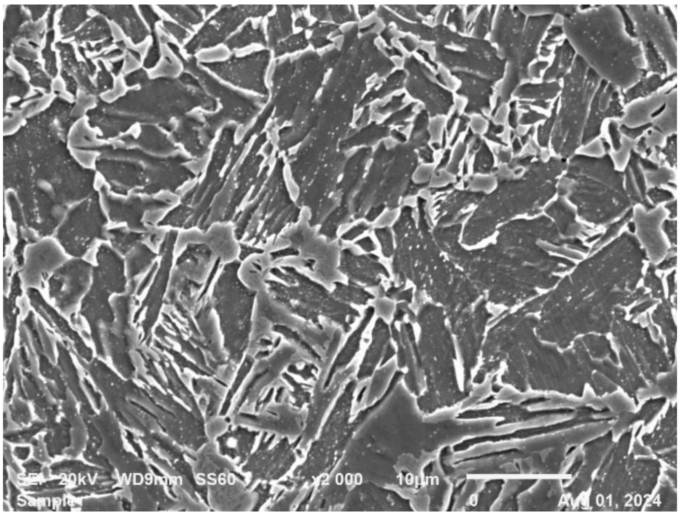

Figure 17. SEM 16 mm specimen x2000, (10μm scale bar), etched in Nital-2, Intercritically Annealed.

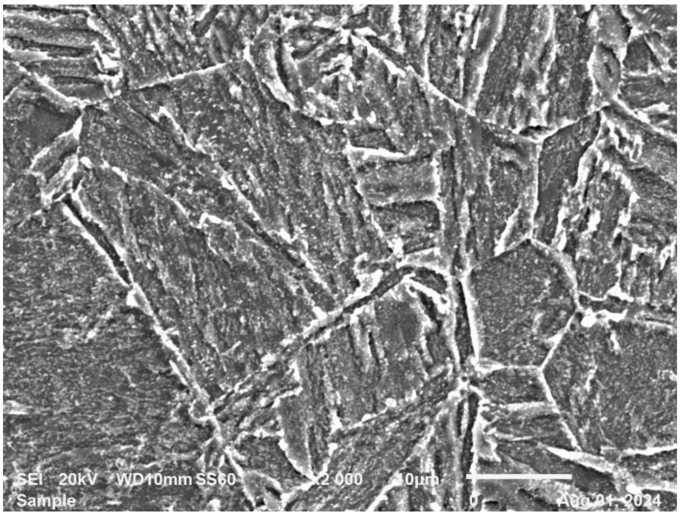

Figure 18. SEM 20 mm specimen x2000, (10μm scale bar), etched in Nital-2, Intercritically Annealed.

5. Conclusion

This study aimed to determine the effects of the proposed two-phase intercritical annealing processing of low-carbon alloy AR400 abrasion-resistant steels typically used to extend the production life of ground-engaging tools and earthmoving machines in the South African mining industry, and to determine whether it can be used as a mechanism to improve the impact energy absorption without significantly reducing or altering the tribological properties. The study hypothesised that the two-phase intercritical annealing process introduced in the methodology could increase the material's impact energy absorption without significantly reducing its hardness. It used a comparative approach to assess the mechanical and material properties of the control specimens, as determined by metallurgical analysis.

The initial spectrometric analyses of the control specimen highlighted the inconsistency found in the alloying element concentration and mechanical properties, especially of major alloying elements responsible for material strength, hardness and corrosion resistance, of the same material grade across different material thicknesses, these inconsistencies confirm the need for regulatory standards governing more than just the material properties, as the lack thereof can correlate to the varying performance and life span of the liner plates and wear packages in mining operations.

The microanalyses revealed a successful increase in bainitic dispersion in the intercritically annealed specimens, regardless of material thickness, which is typically associated with the ability to absorb or withstand impact and maintain structural integrity. However, the change in phase dispersion and the reduction in martensitic phases, resulting from the moderate cooling rate following the Phase 1 heat treatment and oil quenching, significantly reduced the material's hardness, UTS, and yield stress.

The metallurgical examination reveals that the specimens' ductility and impact energy absorption increased to varying degrees. However, the improvement in impact energy absorption was inconsistent across material thickness and too scattered to determine conclusively whether the improvement in impact resistivity was successful. In addition, this improvement was achieved at the expense of material hardness and tribological properties, as material hardness is directly proportional to the material wear rate, contradicting the hypothesis. Although the two-phase intercritical annealing process proposed reduced brittleness and improved impact energy absorption in 20 mm AR400 steels, it is not recommended, in its current iteration, as a mechanism to enhance impact absorption of materials with a thickness below 20 mm without significantly decreasing the material's hardness and tribological properties.

As such, although the proposed process parameters resulted in excessive hardness loss, making the material unsuitable for general mining applications, the substantial improvement in impact energy in 20 mm specimens indicates promise for specific applications where impact resistance is prioritised over heavy-duty wear resistance. Future research recommendations include refining the proposed dual-phase intercritical annealing heat-treatment process and investigating the optimised mechanical properties of abrasion-resistant steels for dual wear and impact applications in the mining industry. Based on the outcome of this research, this proposed future research avenue should focus on improved processing parameters for material thickness, including 20 mm and above, investigating shortened hold times, intermediate quenching temperatures, and tempered martensite approaches as suggested by Cao and Alfirano, to achieve an optimal balance between hardness retention and improved impact resistance.

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Competing Interest Statement

The authors declare no known competing interests that could have influenced the outcome of this study.

Data Availability Statement

All data generated and analysed in this section of the study are included in this article.

Author Contributions

WM: Conceptualisation, material experimentation, data collection and writing; TJ: Supervision and review; LWB and TAA: Data Analysis, supervision and review.

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