Acoustic Emission Monitoring of Ultra-Fast Low Energy Ceramization and Thermite Reaction for Ultra High Strength Steel Processing

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Mahmoud M Abu Elkhier[1], Bakr M Rabeeh[3], S kaitbay[2] and Amir A Abdelsalam[2]

[1] Ph.D. Postgraduate, Benha College of Engineering, Benha University, Egypt
[2] Staff members, Benha College of Engineering, Benha University, Egypt
[3] Professor of Engineering, German University in Cairo (GUC)
Corresponding author: mahmoud.abuelkhier@gmail.com

Abstract: This study explores the application of an ultra-fast ceramization process to enhance the performance of ultra-high-strength steels (UHSS), specifically Mn-Al steel (S-52), crucial for aerospace and automotive applications. The aim is to achieve an optimal balance between strength and ductility through in-situ synthesis of a borate glass ceramic cladding. A thermite-driven fusion reaction, conducted in an induction furnace at 220°C for 30 minutes utilizing a waveguide and powder pack, facilitated the ceramization. The kinetics of this thermochemical process, including alloy segregation and phase transformations, were monitored through acoustic emission and thermal imaging. Microstructural characterization using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) elucidated the reaction mechanism, revealing five sequential stages. The process involved localized melting and alloy segregation, followed by stress-induced two-phase transformation; a thermite reaction resulted in material defragmentation, while twinning and recrystallization contributed to the formation of specific microstructural features, culminating in the development of a robust ceramic/metal interface achieved through both chemical and mechanical bonding. The correlation between these stages and acoustic and thermal data provides a comprehensive understanding of the ceramization kinetics and interface formation. Acoustic emission events characteristics: count, count rate, amplitude, duration, energy, rise time and frequency are captured and correlated with SEM and thermal imaging. Acoustic characterizations are also captured for ultra-high strength steel with total 1,048,576 signal by 2324 microseconds in 30 mins.

Keywords: advanced high performance, Ceramization, Exothermic, Fusion, Functional-graded materials (FGM), Hybrid, Sandwich structure, Acoustic, Thermite, ultra-high strength steel (UHSS).

1. Introduction

The demand for ultrahigh strength steel (UHSS) in industries as: aerospace and automotive is continuously growing; driving the need for innovative and efficient techniques processing. Traditional methods often suffer from high energy consumption and lengthy processing times. This paper investigates the application of ultra-fast low energy ceramization and thermite reactions as a novel approach to UHSS processing,

aiming to achieve rapid and localized material modification. However, the complex and transient nature of these reactions presents significant challenges in real-time monitoring and process control [1]. To address this, we explore the use of Acoustic Emission (AE) monitoring as a non-destructive technique to capture the dynamic events occurring during these ultra-fast reactions which is done count almost 1 million cycle of fast diffusion. This approach enabled the capture and analysis of microsecond-scale events, revealing the synchronized nature of inter-mutual serial processes and the dynamic evolution of phase coupling and shifting. The ability to observe these phenomena in under a minute, compared to the previously researches shown it is required 76 hours which was too much energy and time and production losses compared with the research impact which is done in 30 mins with a perfect diffusion by both the Fe2b tension banding and Feb compression zones which experimentally verified to increase the strength with low melting phase (LMP) to high heat extraction from the boron diffusion happened , marks a significant advancement in the study of ultrafast deformation mechanisms, providing a detailed understanding of localized energy increases and the initiation of elemental segregation. By analyzing the AE signals, it is aimed to gain insights into the reaction kinetics, defect formation, and ultimately, and the quality of the processed UHSS. This research demonstrates the feasibility and effectiveness of AE monitoring in characterizing the ultra-fast low energy ceramization and thermite reaction, providing valuable data for optimizing process parameters and ensuring consistent material properties [2-3], the characterization considers the following definitions as shown in Figure 1:

i. AE Hit: A signal that surpasses the threshold and triggers a system to accumulate data.
ii. Amplitude: The greatest recorded voltage in a waveform, which is directly proportional to the AE energy. The AE amplitude is frequently expressed in millivolts (mV) or decibels (dB).
iii. Duration: The interval of time between the AE signal's initial and final threshold crossings. Usually, the time is stated in microseconds (µ.sec).
iv. Energy: based on the AE data gathering method, energy is defined as the measured area under the rectified signal envelope (MARSE). it is frequently utilized in acoustic emission studies.
v. Rise Time: The amount of time that passes between the AE signal's triggering time and peak. The rising time is often measured in microseconds (µsec), just as the duration.
vi. Counts to Peak: quantity of counts separates the peak amplitude from triggering time beyond threshold.
vii. RMS: Square root of mean of the squares of amplitude of AE impacts is a statistical measure.
viii. AE Counts: The frequency with which the signal surpasses a current threshold. The strikes strength and AE activity are also measured by the number of counts.

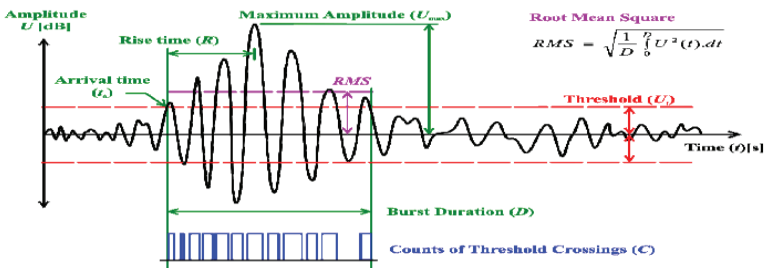


Figure 1. Illustration of AE Signal Parameters [4]

Despite the remarkable properties of UHSS, its processing presents significant challenges. Traditional manufacturing methods, such as conventional hot rolling, quenching and partitioning (Q&P), and thermomechanical processing, often encounter limitations that hinder their scalability and sustainability. These limitations include:

- High Energy Consumption: Traditional heat treatment processes require substantial energy input, leading to increased carbon emissions and operational costs.
- Residual Stresses and Distortion: Intense thermal gradients during processing can induce residual stresses and distortion, compromising the dimensional accuracy and mechanical performance of the final product.
- Environmental Concerns: The use of hazardous chemicals and the generation of waste products in certain processing routes raise environmental concerns.
- Microstructural Heterogeneity: Achieving uniform and refined microstructures across large components remains a challenge, impacting the consistency of mechanical properties.
- Costly processing: many of the heat treatments require long cycle times, and expensive equipment.

The pursuit of advanced manufacturing techniques for Ultra-High Strength Steel (UHSS) has led to the exploration of ultra-fast, low-energy processes, particularly those utilizing ceramization and thermite reactions. These methods hold immense promise for achieving rapid and localized heat treatment, potentially revolutionizing UHSS production by enabling tailored microstructural modifications and surface enhancements [5-8]. However, a significant gap exists in our ability to effectively monitor and control these highly dynamic and exothermic reactions, hindering their consistent and reliable application.

1.1 The Challenges of Ultra-Fast Reaction Monitoring

Ceramization and thermite reactions, by their very nature, occur at extremely high rates, often within milliseconds. This ultra-fast kinetics presents a formidable challenge for traditional monitoring techniques [9-10]. Conventional temperature sensors and imaging systems lack the temporal resolution required to capture the rapid evolution of these reactions. Furthermore, the localized and often confined nature of these processes makes it difficult to obtain comprehensive data on the reaction front propagation, temperature distribution, and phase transformations.

1.2 The Critical Need for Non-Destructive Real-Time Monitoring

Crucially, there is a profound lack of non-destructive, real-time monitoring techniques capable of providing in-situ information about the progression of ceramization and thermite reactions. Current methods often rely on post-process characterization, which provides limited insight into the dynamic evolution of the reaction and its impact on the final UHSS properties. This lack of real-time feedback necessitates reliance on empirical data and iterative process adjustments, leading to inconsistencies and inefficiencies. The need for non-destructive techniques is paramount, as destructive testing halts production and is not useful in a closed loop control system [11].

The Impact on UHSS Property Consistency:

The inability to accurately monitor and control these ultra-fast reactions directly translates to inconsistencies in the resulting UHSS properties. Variations in reaction kinetics, temperature profiles, and phase formations can lead to significant deviations in microstructure, hardness, and residual stress distribution. This lack of process control undermines the reliability and reproducibility of UHSS products, limiting their widespread adoption in critical applications [12-13].

2. Experimental Technique

Chemical composition of as-received Mn-Steel, S- 52N, is established and presented in Table 1.

The UHSS (St-52) alloys of Fe-Mn for the iron percentage of 97.6 wt. % and C 0.2 wt. % have been surface diffusion for Boronizing and ceramization process using electrical resistance furnace. The electrical coils in the electrical resistance furnace Nabertherm were made of Kanthal electrical coils. The bottom holding tray graphite was used to Hold the specimen of dimensions according to ASTM D790

L 125 mm x W 12.7 mm x T 3.2 mm per the ASTM standard of Fe-Mn alloy each to produce the Ceramized layer diffused on St-52. The specimen was heated up to 220ºC as shown in figure 2-a for borat glass diffusion and the surface layer which led to be transferred into Ceramized layer of thickness of 6.5 mm approximately. The K type thermocouple was used to measure the temperature during diffusion in ˚C. by a thermal camera was used for detection of that application to detect the diffusion. Similarly, the acoustic determination happened with correspondence that have a significant result will be shown later. The cutting length of the samples have been cut by CNC cutting machine with high precision according to ASTM D790. The optimized controlled temperature of 150 + 70 ˚C was employed for ceramization process. The speed, energy and counts of diffusion of the ceramization process was measured by acoustic emission (AE) system. The samples for microstructure from the diffusion samples (outer, inner and center region) and ceramization – borat layer shown the banding which happened on the grain boundaries defining the (tension and compression regions) with the segregation of atoms on the surface. Which cut at different places as per the ASTM standards. All samples were metallographically prepared according to ASTM E3-11 to achieve a mirror like finish. The procedure included sequential grinding with silicon carbide papers of grits ranging from 60 µm to 1200 µm, followed by polishing with diamond suspensions from 6 µm to 3 µm to 1 µm and final polishing using colloidal silica. The diamond paste and spray respectively used for alumina and diamond polishing using velvet cloth.

Table 1. Chemical composition of Mn- Steel (S-52N) wt. %

Element	C	Si	Mn	P	S	Cr	Ni	Mo	Al
%	0.209	0.209	1.3	0.0078	0.011	0.131	0.122	0.0043	0.029
Element	Co	Cu	Nb	Ti	V	W	Pb	B	Mg
%	0.027	0.247	<0.004	<0.001	0.012	<0.010	<0.003	0.0034	<0.001
Element	Ta	B	Sn	Zn	As	Ce	Zr	Fe balance	
%	0.018	0.00114	0.025	0.0051	0.027	0.0069	0.0036	97.6	

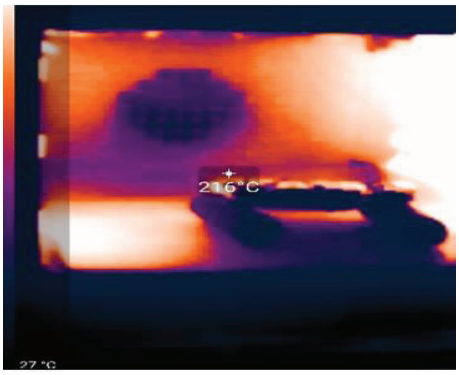


Figure 2-a. Thermal processing medium during ceramization of steel 52 at 220 °C

The speed of the disc was 250 rpm, and ten-minute polishing was done on each sample. The polished samples of Ceramized of UHSS St-52 etched according to ASTM E407 are for 6 seconds using (5 ml HNO3, 3 ml HCl and 2 ml HF 40%) Aqua Regia etchant which it is very strong acid which helps in showing the very tough ceramic layer that happened on the surface showing the localized melting adopted with the segregation happened. The microstructural investigations were carried out using Struers Labopress-1, Zeiss Mrc5 optical metallurgical microscope and for Scanning electron microscope was conducted by FEG Emera FEI Quanta.

The Acoustic Emission (AE) monitoring relies on a specialized data acquisition system to capture and interpret material behavior. At its core, highly sensitive sensors detect minute vibrations, which are then strengthened by preamplifiers and refined through filtering to eliminate extraneous noise. The system digitizes these signals using analog-to-digital converters (ADCs), the signal was obtained using an AE sensor (model: PKWDI) having an operational frequency range of 200–850 kHz. The sensor was positioned at the valve/cylinder cover and used super glue to adhere it tightly to the surface. The full AE hit is provided by a single-channel AE data acquisition device (model: USB AE Node) with AEwinTM software. Time-based features, such as waveforms, were utilized to capture the AE signal and extract the AE parameters. enabling computer-based analysis. Dedicated software then dissects the resulting waveforms, extracting vital metrics like signal strength, released energy, frequency components, event counts, and rise time [14-15].

This research explores boron diffusion into steel Grade 52 using acoustic emission (AE) to identify optimal processing parameters (temperature and time). Unlike previous studies that report diffusion at 500-700°C, this approach investigates achieving similar results at lower temperatures and faster processing times. The winTM AE system, equipped with GI40lG40 sensors, enables real-time monitoring of the diffusion process. The system's high sampling rate (2324 microseconds/count), multi-channel capability, and advanced filtering (200–850kHz high-pass, 1-2.5MHz digital) allow for accurate and reliable data collection. Key benefits of this system include rapid data transmission, flexible expansion, stable performance, precise diffusion depth determination, and suitability for continuous monitoring. Data analysis is performed using Minitab software [16-17].
A digital filter, adjustable from 1 to 2.5 MHz, refines the signal. This measurement system offers high-speed data transmission, flexible expansion, stable performance, precise diffusion depth localization, and suitability for continuous monitoring as shown in figure 2-b.

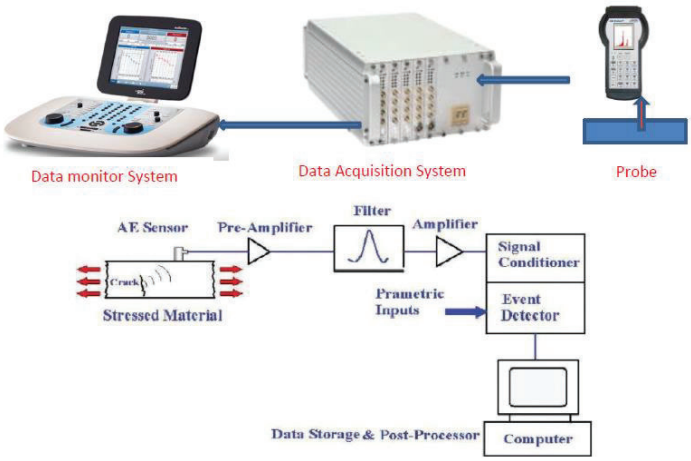


Figure 2-b. Diagram showing the measuring of acoustic system

These parameters offer critical information about the nature and intensity of internal events. Comprehensive data storage ensures that all information is recorded for subsequent evaluation. In essence, this advanced system functions as a highly perceptive listener, transforming the sounds of material activity into actionable data for analysis and interpretation [18].

3. Experimental Results (Physical and microstructural characterization)

The work examined ST 52 steel samples produced by ceramization method: Ultra-fast surface diffusion and Boronization. The density of the steel-52 was found to be approximately 7.85 g/cm³, while the diffused cast steel was slightly denser at 7.88 g/cm³. This small difference indicates that both methods resulted in consistent material density. Microscopic images revealed the internal structure of the steel with the surface coating on both tension and compression edges. The Ceramized samples showed a phase distribution influenced by the rapid temperature during the process, around 10 °C/s.

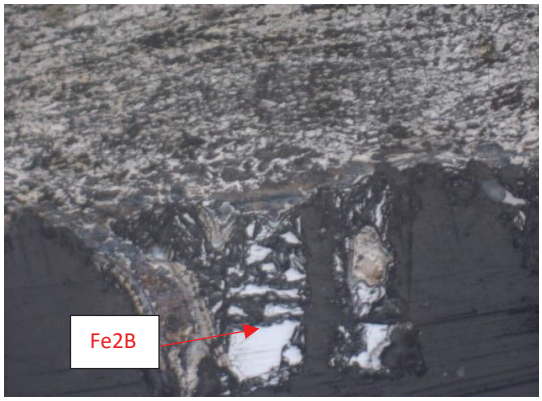


Figure 3. ST - 52 10x at tension

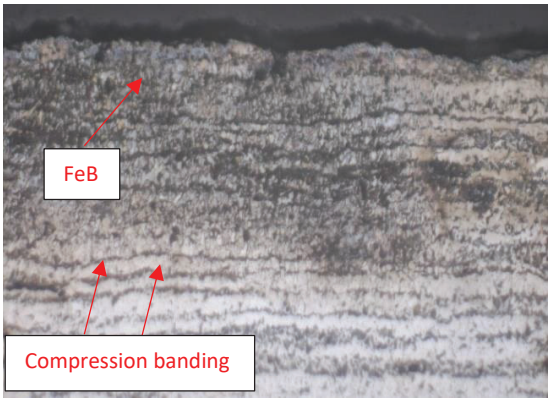


Figure 4. ST - 52 10x at compression

The above figures (3 – 4) show distinct layers formed by the diffusion of borate glass, Fe2B, and boric acid into steel 52 under tension. Showing homogeneous crystalline metals which exhibit ultra-high as the strain to failure in a uniaxial tension zone compared to figure 4 showing the compression banding to stabilize the stress mismatch; Moreover, the irregular interface and contrasting regions suggest a complex interaction zone with potential ceramic phases and thermite reaction products. This microstructure likely contributes to the ultra-high strength of the processed steel.

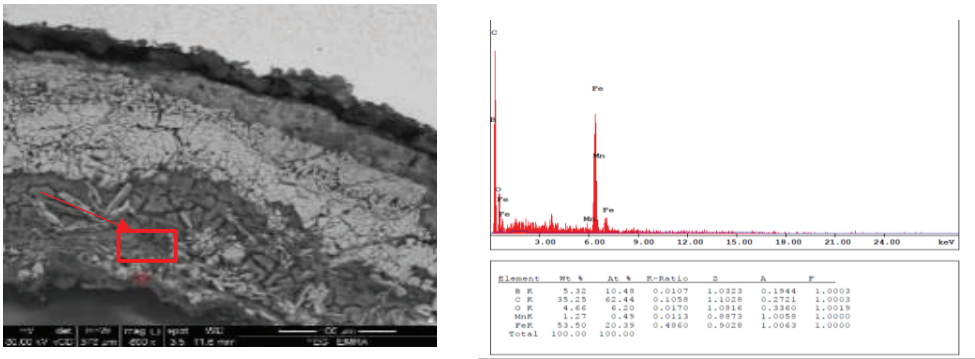
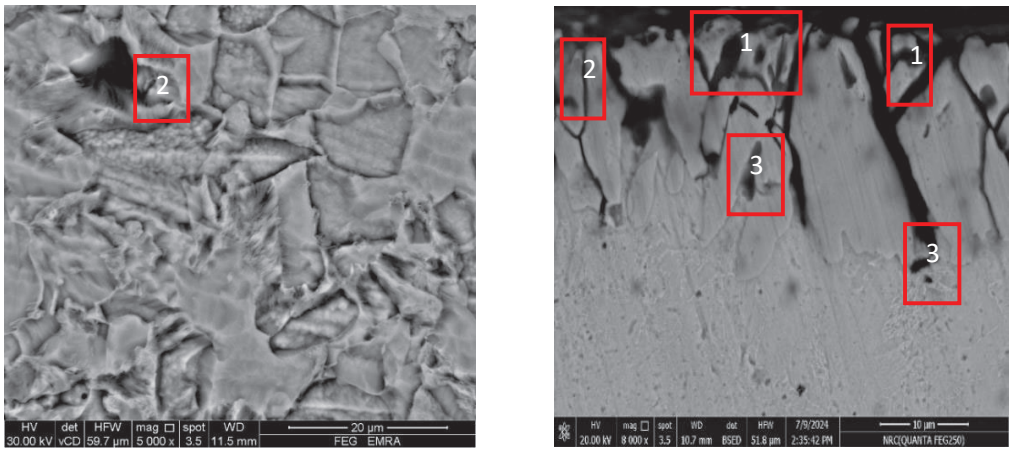


Figure 5. The EDX presents the diffusion of Boron by 5% causing defragmentation Fe2B segregation and Localized melting with alloy, the results are verified by output of high-resolution electron microscopy



(a) **(b)**

Figure 6a-b. Presents the scanning electron microscopy of ceramized Mn-alloy steel (S-52N)

In figure (a) the process carried out at 180°C for 30 min with Boron Mix showing the particularly during defragmentation (1), while the figure (b) shows the Fe2B segregation (2) and the localized melting with alloy segregation with stress mismatch (3).
The test followed by The Acoustic signals which exposure takes approximately 0.002324 seconds per count of total 1,048,576 signal by 2324 microseconds as shown in figure 6 which causes an ultra-fast surface diffusion to be in 30 mins compared to the historical diffusion which done almost in 76 hrs.

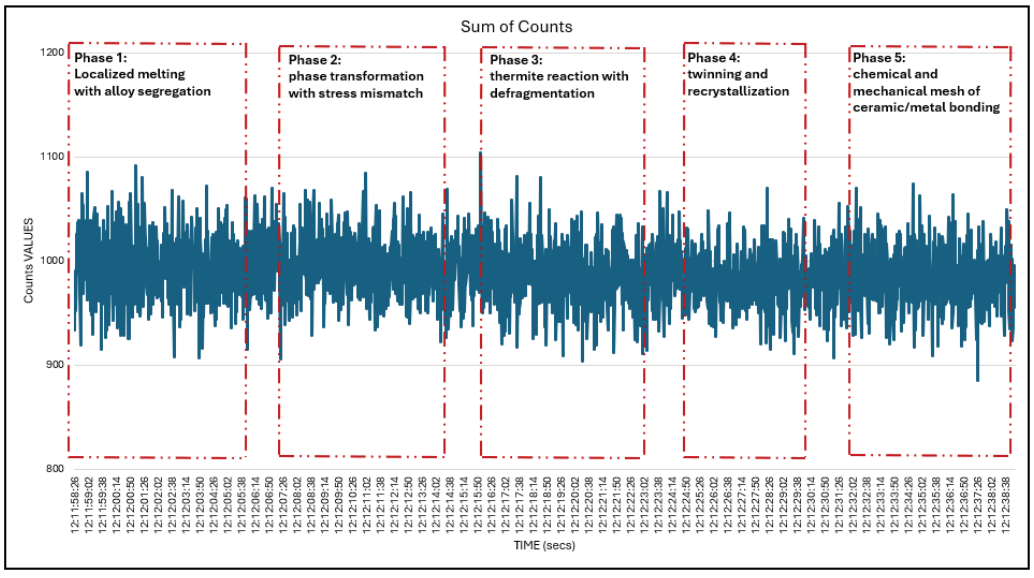


Figure 7. Energy minimization and the dynamic nature of ultrafast events, showing how the 'Sum of Counts' varies with each stage of the process in very high-speed diffusion with the events of the peaks

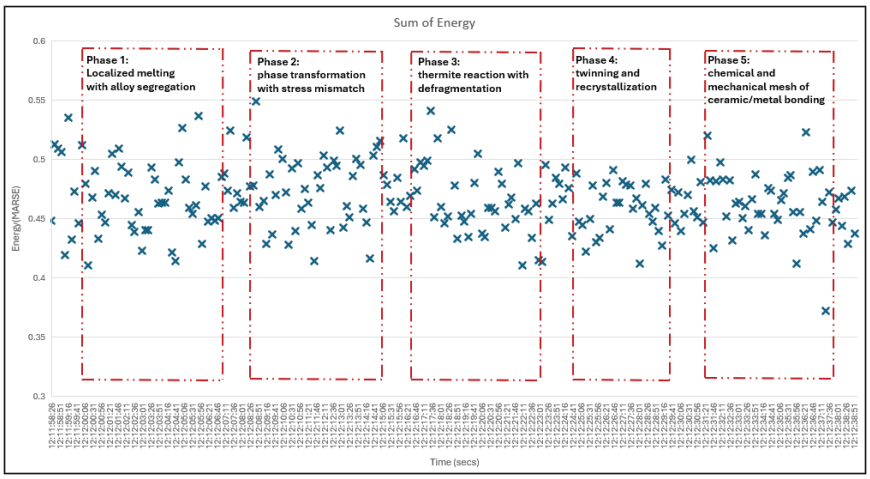


Figure 8. Scattered localized sum of melting energy fluctuations across five phases

The shown recorded phases reflect dynamic processes like melting, phase transformation, and thermite reaction. These fluctuations are correlated with microstructural changes, offer insights into energy balance and kinetics for process optimization.

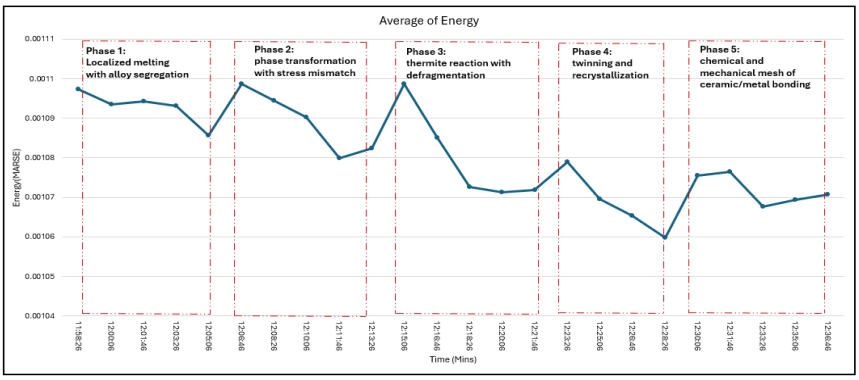


Figure 9. Average distinct energy fluctuations across the five phases with alloy segregation

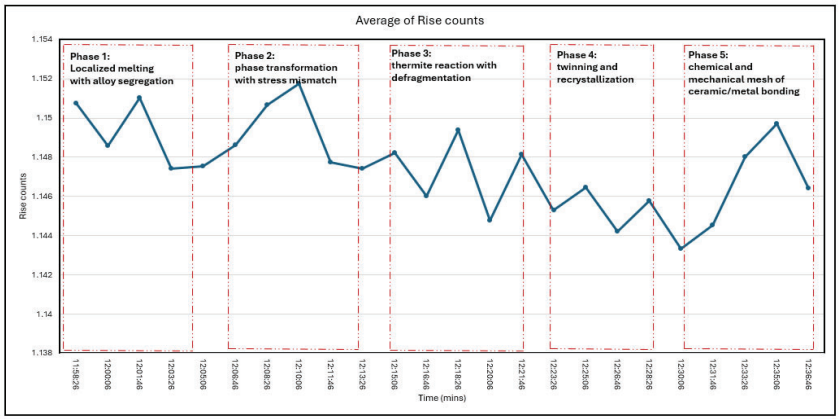


Figure 10. Localized average of energy increase counts recorded at atoms and grain boundaries

The records shown in figure 10 are visualized through the bulk thermal microscope, correlate with the fluctuations in both graphs, particularly during defragmentation and Fe2B segregation in Phases 3 and 4. These fluctuations reflect the energy involved in crystal contraction, dipole formation, and the release of free grains into the liquid carbon phase at 150 + 70 degrees, as depicted in the 'Rise Counts' graph.

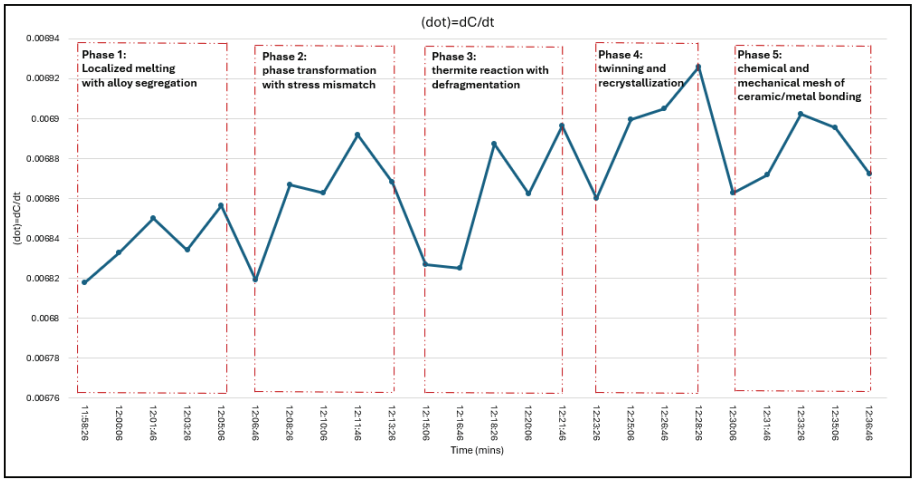


Figure 11. Rate of acoustic emission (dC/dt) across five distinct phases of material transformation.

The fluctuations in acoustic emission rate dC/dt presents the microstructural change rates or crack growth, it also indicates dynamic activity during each phase, with notable peaks suggesting heightened transformation events

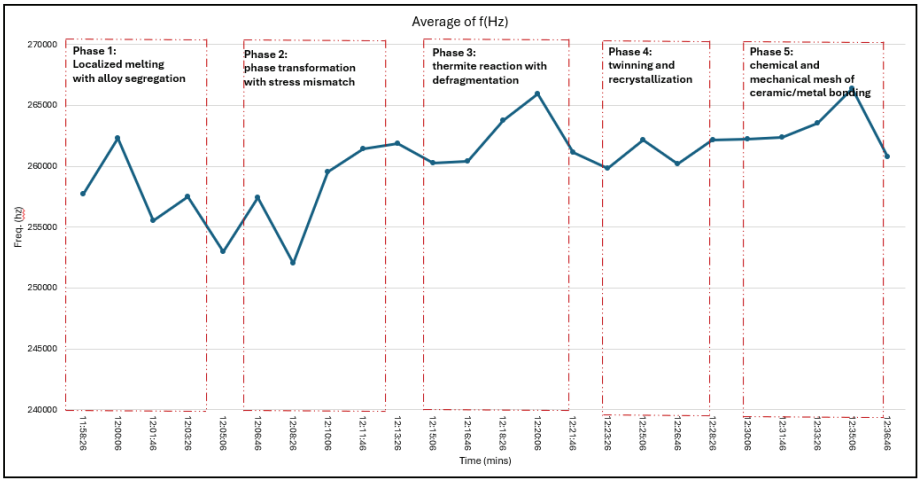


Figure 12. The graph shows a notable peak in Phase 3, the peak indicate the increased energy due to fragmentation.

The consistent event characteristics, rate of increase, and slope across phases suggest inter-mutual serials and phase coupling, despite energy increases during meshing.

4. Conclusion

In conclusion, this research delved into the complex interplay of energy minimization, time constraints, and ultrafast deformation mechanisms within crystalline materials. Utilizing 180-degree tracking via acoustic, thermal, and optical EDX techniques, coupled with DFT modeling, despite its energy demands due to fragmentation. The work proves the obtained successfully characterized events occurring in microseconds. On other hand, the analysis revealed consistent event characteristics, including rate of increase, event occurrence, and slope, demonstrating the synchronized nature of inter mutual serial processes. Phase coupling and shifting were observed, alongside the energy increase associated with meshing. We achieved ultrafast observation, capturing events in under a minute that previously required 76 hours, revealing localized energy increases at atomic and grain boundary levels, visualized through bulk thermal microscopy. This research also documented fusion during crystal defragmentation, the formation of dipoles, and the commencement of Fe2B segregation, where tension led to a decrease in the lattice parameter at the nanoscale. Furthermore, crystal contraction was shown to localize tension, releasing free grains into a liquid carbon phase at 150 + 70 degrees. These findings underscore the importance of understanding the intricate balance between energy minimization and time during ultrafast material transformations by monitoring the 1,048,576 signals from the beginning with no ramping up time, providing a foundation for future advancements in materials science.

This study successfully demonstrated the effectiveness of acoustic emission monitoring, utilizing a specialized data acquisition system with a sampling rate of 2324 microseconds/count and advanced filtering (200-850kHz high-pass, 1-2.5MHz digital), to optimize boron diffusion in steel at lower temperatures than the traditionally reported 500-700°C for 76 hours, offering significant potential for enhanced processing efficiency which causes an ultra-fast surface diffusion to be in 30 mins compared to the historical diffusion.

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