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Methodology of the Planned Interval Test for Metal Corrosion in Phase Change Materials

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Abstract

Phase Change Materials (PCMs) represent a promising technology for passive thermal management, thereby enhancing the energy efficiency of buildings. However, their widespread implementation is limited by the corrosion risk associated with metallic encapsulation materials when in contact with PCMs, which compromises their long-term stability and integrity. This contribution describes in detail and introduces the Planned Interval Test (PIT) methodology as a standardized and comprehensive approach for the systematic analysis of metal corrosion processes within dynamic PCM systems. Unlike conventional static tests, PIT enables the precise monitoring of corrosion kinetics as a function of total exposure time and the number of thermal cycles, while also identifying individual degradation phases. This information is crucial for predicting durability and designing robust encapsulation systems. The primary objective of this work is to validate a procedure for the accurate evaluation of metallic corrosion resistance, thereby supporting the development of safer and more durable PCM solutions for sustainable construction and mitigating the environmental burden resulting from premature material failure.

Keywords: phase change materials, planned interval test, metal corrosion, encapsulation

1 Introduction

Current initiatives focused on sustainable development and the reduction of energy consumption in the construction sector emphasize the development of innovative materials [1]. In this context, Phase Change Materials (PCMs) play an increasingly critical role in the concept of passive thermal management within building structures [2]. Due to their unique capacity to store and release a significant amount of latent heat during isothermal phase transitions (melting and solidification), internal building temperatures can be efficiently regulated. This leads to enhanced thermal comfort and a potential reduction in heating and cooling energy demands [3]. Despite their promising potential, the widespread implementation of PCMs in the construction sector faces major obstacles [4], particularly concerning the compatibility and long-term integrity of encapsulation materials. PCMs are typically enclosed in containers to prevent leakage, avoid contamination of surrounding materials, and ensure their long-term stability.

Metals (such as aluminum, copper, or various steels) are often chosen for these containers due to their excellent thermal conductivity, mechanical strength, and relative ease of processing [5]. The issue of corrosion of metallic enclosures in contact with PCMs is a subject of intense research [6], with the most frequent failure mode, which critically jeopardizes system reliability, being the leakage (oozing) of the PCM. Multiple studies have confirmed that corrosive interactions can lead to the premature destruction of PCM systems. For instance, a comprehensive review emphasizes that the interaction of PCMs with surrounding packaging materials can lead to reduced performance and durability [7]. Research systematically examined the corrosion of various metals (e.g., aluminum, carbon steel, stainless steel, copper) in contact with organic and inorganic PCMs under simulated cyclic conditions, finding significant corrosion during long-term exposure and higher operating temperatures, which can cause perforation of the containment and subsequent PCM leakage [8]. Similarly, studies focused on the corrosion compatibility of fatty acid esters and identified that even seemingly inert PCMs can induce corrosion in metals such as copper and aluminum [9]. Other investigations confirmed that the failure of container walls, whether due to corrosion or mechanical damage, or the influence of minor changes in pH and moisture, is the main cause of PCM leakage and reduced efficiency [10, 11]. Review papers summarize the causes of corrosion (presence of oxygen, moisture, impurities, electrochemical potentials) and mechanisms (e.g., pitting, crevice corrosion) and consistently point to instances where the failure of containment materials led to undesirable PCM leakage and subsequent degradation of the entire thermal storage system [12]. The degradation of the enclosure not only reduces the system's thermal capacity and functionality but can also potentially damage surrounding building structures and compromise occupant health [13, 14]. Therefore, addressing the corrosive compatibility of PCMs with encapsulation materials is a priority to ensure the durability and reliability of these innovative building solutions.

2 Planned interval test (PIT)

Material corrosion is a critical factor affecting the reliability and lifespan of technological systems, particularly in construction, where it can lead to premature component failure and increased maintenance costs [15]. Corrosion processes in dynamic environments, such as those involving PCMs, are especially complex. In these systems, metals are subjected to cyclic temperature changes and phase transitions, creating unique corrosive conditions that differ significantly from those in typical static environments [16,17]. To ensure the reliability of PCM technologies, it is essential to employ accurate methodologies for corrosion assessment under these dynamic conditions. Traditional immersion tests with a single long exposure are inadequate because they fail to provide a detailed insight into the corrosion kinetics as a function of exposure time and the number of cycles [18]. These standard methods, designed for stable conditions, often only yield an average corrosion rate [19], thereby losing valuable information about initial acceleration, stabilization, or other changes in rate over time. For this reason, the PIT methodology (also known as the interval corrosion test) is considered an optimal approach [20]. PIT enables the monitoring of corrosion progress over discrete time intervals. Instead of measuring a single final weight loss, samples are withdrawn at different phases of the test, allowing for a deeper understanding of the kinetics [21]. The method reveals whether the corrosion rate accelerates over time (e.g., due to the growth of corrosion products), slows down (thanks to passivation layers), or fluctuates based on the simulated phase transition cycles, which is a key aspect for phase change materials [22,23]. The paper should contain a description of methods and research approach in sufficient detail to enable other researchers to perform similar analyses or link their research to the reported results. Well-established methods can be briefly elaborated and adequately cited.

3 Methodology

Achieving reliable and reproducible results when analyzing metal corrosion in dynamic environments demands the application of a precisely defined experimental protocol. While no specific international standard has yet been adopted for the PIT methodology in the context of PCMs, this procedure is fundamentally based on the core principles of general corrosion testing. Specifically, the methodology draws upon the recommendations of standards such as ASTM G31 for immersion testing and ASTM G1 for sample preparation and cleaning procedures. These existing standards form the necessary basis for proper sample preparation, exposure, effective cleaning, and accurate mass loss measurement, all of which are essential components of the PIT [18,24].
Table 1 summarizes a series of steps designed to ensure the accurate measurement of mass loss and visual changes in the tested samples, which are key indicators of the rate and character of corrosion. This comprehensive procedure allows for detailed monitoring of corrosion kinetics and includes the following phases, which are repeated for each testing interval [25].

Table 1: Experimental procedure for the planned interval test

Step	Experiment Procedure
1	selection and cleaning of samples
2	marking and numbering of samples
3	weighing and measuring the dimensions of the samples
4	recording the original surface appearance of the samples using a microscope
5	re-cleaning and degreasing of the samples
6	immersion of the samples into the testing material in a hermetically sealed container
7	removal of the samples at given time intervals
8	cleaning of the samples with water and checking for changes in surface appearance
9	recording the changes in the surface appearance of the samples using a microscope
10	removal of corrosion from the samples using a cleaning sponge
11	weighing the samples after corrosion removal
12	evaluation of the test based on the calculation of the corrosion rate

3.1 Sample preparation

For testing purposes, samples of representative dimensions and shape are prepared, designed to minimize the influence of edge effects and allow for accurate mass loss determination. Test materials are selected from metals typical for PCM encapsulation (e.g., various types of aluminum, copper, or steel). Before exposure, the sample surfaces are treated using standardized grinding and polishing methods to achieve uniform and comparable surface roughness. The final preparation before weighing includes thorough degreasing in a suitable solvent, drying, and subsequent determination of the initial mass with high precision, ideally to 0.1 mg, which is critical for subsequent corrosion kinetics calculations [25].
To better understand the practical implementation of the PIT methodology and to illustrate the initial phases of the experiment, Figure 1 shows the key stages of test sample preparation, including their placement in the testing containers, immersion in the phase change materials, and storage in the environmental chamber.

a) b) c)

Figure 1: Preparation of samples for the PIT: a) prepared metallic samples before immersion; b) metallic samples immersed in PCM in sealed containers; c) placement of samples in the environmental chamber

3.2 Realization of the PIT

After the hermetic sealing of the containers, the test sample sets are transferred to a climate chamber and exposed to precisely controlled thermal cycles, which accurately simulate the PCM phase transition and the dynamic operating conditions typical for construction applications. The 24-hour cycle model utilized for the test, consisting of four distinct phases (heating, high-temperature hold at 35 °C, cooling, and low-temperature hold at 15 °C, is illustrated in Figure 2. A key aspect of the PIT methodology lies in the regular withdrawal of identical samples at precisely predefined time intervals or after a specified number of cycles [26, 27]. The determination of these intervals is a critical strategic element selected to capture the full corrosion kinetic curve. This approach addresses the known limitations of short-term corrosion tests (e.g., 25-day tests), which are often insufficient for materials forming passive films. For these materials, prolonged testing is necessary to detect localized attack or film breakdown, making long-period tests considerably more realistic. The planned-interval test is, therefore, recognized in corrosion guides (e.g., ASTM G31/NACE TM0169) as the recommended procedure for evaluating the effect of time on corrosion. Accordingly, the strategic selection of intervals for this methodology must be structured to achieve this explicit goal: it requires Initial Shorter Intervals designed to capture the formation of the protective passive layer, followed by Subsequent Extended Intervals, which are crucial for monitoring long-term stability and identifying critical failure points where cyclic thermal stress may cause a breakdown of the passive layer [18].

In our specific test setup, we utilized withdrawal intervals at 4, 12, 16, and 20 weeks of exposure. This sequence ensures that the early passivation kinetics are captured, followed by monitoring long-term stability at intermediate and extended durations under dynamic thermal cycling. This comprehensive approach yields robust data essential for reliable, long-term durability prediction.

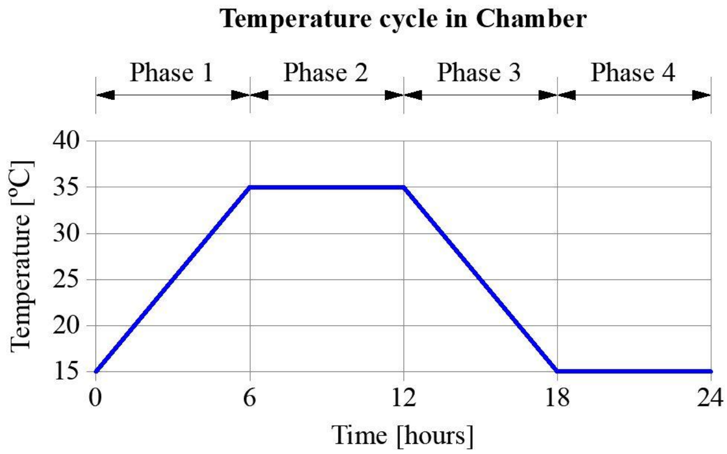

Figure 2: Temperature cycle in Chamber

Each withdrawn sample then undergoes a rigorous process of cleaning corrosion products, precise weighing, and visual inspection. This detailed approach is essential for obtaining kinetic data on mass loss as a function of time and cycles. This data provides a comprehensive view of the corrosion process, including the identification of its initial phases and any changes in corrosion rate. The data processed in this manner is crucial for a deep analysis of material compatibility and the subsequent optimization of long-term testing conditions [28].

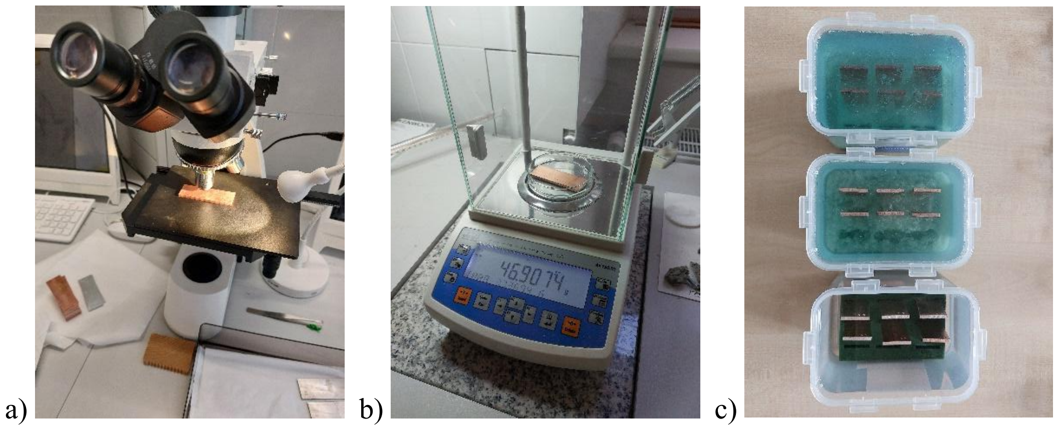

Figure 3: PIT progress: a) monitoring corrosion under a microscope; b) weighing samples after a time interval; c) copper corrosion during the PIT.

3.3 Evaluation of the PIT

Upon the completion of each defined time interval (or reaching a predetermined number of cycles), selected samples are purposefully withdrawn from the test exposure. This is followed by the critical cleaning process, aimed at the complete removal of corrosion products and residual PCM. This phase requires utmost caution to ensure the corrosion layer is removed without damaging the underlying metallic substrate. For this purpose, specific chemical solutions are used (e.g., inhibited acids, which are defined by standards for cleaning corrosion products) or, if necessary, gentle mechanical cleaning, always in strict adherence to the procedure set out in the ASTM G1 standard. After successful cleaning, the samples are thoroughly rinsed, dried, and subsequently precisely weighed. The determined mass loss (Δm) is then used as the primary data point for calculating the corrosion rate. The corrosion rate (CR) value is commonly used for the quantitative assessment of corrosion damage. The calculation of the corrosion rate, based on mass loss, is performed using the following relationship [24]:

$$CR = \mathrm{k} * \left(\frac{\mathrm{W}}{\mathrm{d} * \mathrm{A} * \mathrm{t}}\right) \tag{1}$$

where:
CR – Corrosion Rate (mpy) - millimeters per year
k – constant $3.45 * 10^6$
W – weight loss (g)
d – density of the corroded material (g/cm^3)
A – area of the sample (cm^2)
t – exposure time (h)

4 Discussion

The critical importance of the PIT lies in its unique ability to move beyond the limits of static tests, which only provide a time-averaged corrosion rate, to generate interpretable kinetic models. These models, based on mass loss dependency on the number of thermal cycles, are essential for creating reliable predictive models and setting adequate safety margins for decades of system operation [29, 30].

In the dynamic environment of PCMs, the PIT methodology is invaluable for identifying corrosion phase transitions. PIT can reveal the critical failure mode where a metal's initial passive layer, which may appear stable in a static test, is compromised after repeated thermo-mechanical stress. This stress, induced by the cyclic volume changes of the PCM, often leads to a sudden re-acceleration of mass loss, which is indicative of localized damage like crevice corrosion or a breakdown in surface integrity. Detecting these critical, time-dependent failures is paramount for mitigating the risk of premature PCM leakage [31].

The implementation of this complex, yet transparent, procedure contributes significantly to the standardization of testing within the PCM industry. While the PIT methodology is more demanding on time and resources compared to simpler methods, its capacity to eliminate the risk of field failure and facilitate precise material optimization (e.g., selecting ideal alloys or inhibitors) makes it an essential tool. The robust data derived from PIT provides manufacturers and structural engineers with the necessary confidence for the successful, mass commercial deployment of PCM technologies [32].

Although this contribution focuses on detailing and standardizing the procedure, the described methodology serves as the essential framework for generating the kinetic data required for the robust long-term durability predictions, which static tests are incapable of providing.

5 Conclusion

This study introduced and detailed the Planned Interval Test (PIT) methodology as a comprehensive and highly effective tool for evaluating the corrosion compatibility of metals in dynamic PCM systems. The PIT method was shown to fundamentally overcome the limitations of traditional static immersion tests, which are incapable of capturing the complex corrosion mechanisms induced by cyclic temperature changes and PCM phase transitions. The key contribution of PIT lies in its unique ability to monitor and analyze corrosion kinetics over time and thereby identify all phases of degradation. This information is essential for accurately predicting the long-term durability of encapsulation materials. In conclusion, this contribution affirms the critical importance of detailed corrosion analysis for the development of robust and durable PCM systems, which are a cornerstone of sustainable and energy-efficient construction. The PIT methodology is thus validated as an invaluable tool for designing reliable PCM solutions and for reducing the environmental burden resulting from premature material failure. In conclusion, this contribution affirms the critical importance of detailed corrosion analysis for the development of robust and durable PCM systems, which are a cornerstone of sustainable and energy-efficient construction. While this paper focused on the standardization and detailed description of the PIT methodology, the experimental validation of this method across various PCM/metal combinations will be the subject of our subsequent research.

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