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A NOVEL STEPWISE THERMAL PROTOCOL FOR SESSILE DROP WETTING TESTS OF LIQUID LEAD AND REFRACTORY METALS FOR NUCLEAR APPLICATIONS

By applying the sessile drop method, a novel procedure for testing the physicochemical interaction between liquid metals and refractory solid metals used in nuclear reactors was developed. It involves two methodological approaches: (1) in situ surface purification of liquid metal drops from its native oxide film directly in a vacuum chamber, by squeezing liquid metal from a capillary placed above the tested substrate, and (2) cyclical and stepped temperature change involving stepwise heating from 400 to 600°C, followed by cooling back to 400°C using two separate protocols (i) a single heating cycle from 400 to 600°C, followed by gradual cooling to 400°C, and (ii) a repetitive cycle involving heating from 400 to 600°C, cooling to 400°C, reheating directly to 600°C, and final cooling to 400°C.

Wettability tests of selected refractory metal substrates (Me = Mo, Ta, and Ti) with liquid Pb using the sessile drop method and a new procedure showed that the wetting kinetics and contact angle of Pb/Me couples are significantly affected by temperature, but only during the gradual heating up to a maximum of 600°C. During the subsequent steps to final cool down to 400°C, the contact angle for all tested Pb/Me couples remains unchanged.

Measurement discrepancies were observed depending on the capillary materials used (graphite vs. alumina) and surface conditions (polished vs. ground). The results indicate that under the investigated conditions, liquid Pb does not wet the Mo substrate (with increase in temperature, $\theta_{Pb/Mo}$ ~ 115-100°), shows weak wetting on the Ta substrate ($\theta_{Pb/Ta}$ ~ 87-84°), better wetting on polished Ti ($\theta_{Pb/Ti}$ = 30° and $\theta_{Pb/Ti}$ = 67°, with graphite and alumina capillaries, respectively), and good wetting on rough, ground, and unpolished Ti substrate ($\theta_{Pb/Ti\_unpolished}$ = 28°).

Keywords: Metal coolants; Nuclear reactors; Liquid Lead; Refractory metals; Sessile drop method

1. Introduction

Nuclear energy remains a key component of the global strategy to achieve a low-carbon energy future. To ensure the safety and efficiency of next-generation reactors, advanced cooling systems and high-performance structural materials are required. Among various coolant options (TABLE 1), liquid lead (Pb) and lead-based alloys (e.g., Pb-Bi, Pb-Li) have garnered considerable interest due to their high boiling point (1737°C for Pb), low vapor pressure, excellent thermal conductivity, and chemical inertness in contact with air and water [1-2,6]. In addition to their favorable thermophysical properties, Pb-based coolants offer significant advantages in terms of neutron economy. Lead has a low neutron absorption cross-section and excellent gamma shielding capabilities, making it particularly attractive for fast-spectrum reactors [1,3]. Furthermore, the use of lead and its alloys, particularly Lead-Bismuth Eutectic (LBE), offers significant advantages for nuclear reactor safety. One of the key benefits is the ability to support passive safety features, such as natural convection cooling, which becomes critical in emergency scenarios where forced circulation may be compromised [1-7]. These thermophysical and chemical properties make liquid lead a highly promising coolant candidate, especially for Generation IV reactor designs, including Lead-cooled Fast Reactors (LFRs).

Despite their advantages, the interaction between liquid metal coolants and structural materials, particularly the wetting behavior, remains a significant challenge. Among other factors, wetting plays a critical role in the cooling performance of nuclear reactors. On one hand, good wetting enhances heat transfer efficiency by increasing the contact area between the heat source and the coolant, thereby allowing more effective heat removal. On the other hand, wetting can contribute to one

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TABLE 1

Main characteristics of coolants in nuclear reactors [2]

Coolant	Atomic mass [g/mol]	Relative moderating power	Neutron absorption cross-section (1 MeV) [mbarn]	Neutron scattering cross-sections [barn]	Melting point [°C]	Boiling point [°C]	Chemical reactivity (with air and water)
Pb	207	1	6.001	6.4	327	1737	Inert
LBE	208	0.82	1.492	6.9	125	1670	Inert
Na	23	1.8	0.230	3.2	98	883	Highly reactive
H_2O	18	421	0.1056	3.5	0	100	Inert
D_2O	20	49	0.0002115	2.6	0	100	Inert
He	2	0.27	0.007953	3.7	—	–269	Inert

of the most severe degradation mechanisms of structural materials, i.e., Liquid Metal Embrittlement (LME), a phenomenon in which otherwise ductile metals become brittle upon contact with certain liquid metals [8]. This embrittlement is often initiated by wetting and subsequent penetration of the liquid metal along grain boundaries, leading to intergranular fracture and loss of mechanical integrity [8-11].

Therefore, a better understanding of the wetting behavior of liquid Pb-based coolants in contact with different metallic structural materials is a key factor in assessing their compatibility and long-term performance. However, experimental data on wetting properties of liquid metals are often inconsistent and difficult to interpret due to the influence of multiple variables, including testing conditions (temperature, atmosphere, methods and procedures), material chemistry and impurities, and sample preparation (surface oxidation, roughness) [12-15]. For example, early studies by Naidich et al. [12] and later by Protsenko et al. [13-15] demonstrated that the presence of oxide films on both the Pb drop and the metal substrate can significantly alter the measured contact angle and wetting kinetics. Recently, Giuranno et al. [16] conducted a comprehensive study on the wetting behavior of liquid lead on various steel substrates considered for Generation IV nuclear reactors, including T91, AISI 316L, and oxide dispersion-strengthened (ODS) steels such as ODS-12Cr and ODS-14Cr. Using the sessile drop method at a temperature range of 400-820°C and under different oxygen partial pressures, they observed that low oxygen environments promote dissolution of steel components into liquid Pb, while oxidizing conditions favor the formation of protective oxide films that inhibit interfacial degradation. Notably, a transition from non-wetting to wetting behavior was identified around 750°C, with contact angles decreasing significantly at higher temperatures. These findings underscore the importance of in situ oxide removal and controlled testing environments, such as testing atmosphere and temperature, in determining wetting kinetics and interfacial stability, providing valuable insights that support the methodology adopted in the present study.

In addition, previous research has shown that oxide stability and interfacial reactions can vary strongly depending on the substrate material. Heinzel et al. [17] demonstrated that Ti_3SiC_2 forms a protective TiO_2 scale when exposed to Pb or Pb-Bi alloys containing oxygen, which prevents dissolution and ensures long-term stability. Opposite, Mohd and Pletcher [18] reported that effective deposition of PbO_2 on Ti requires careful surface preparation and a layered electrode strategy, again highlighting the central role of substrate condition in Pb/Ti systems. Protsenko et al. [19] further emphasized that removing or reducing surface oxides is crucial for Pb wetting on refractory metals such as tungsten, and that even in systems without bulk miscibility, strong surface interactions can lead to improved wettability.

At the nanoscale, Pb displays additional complex interfacial behavior; Krupski [20] showed that Pb layers on Mo(110) exhibit quantum size effects that stabilize certain island heights, while Logacheva et al. [21] observed the formation of crystalline $PbTiO_3$ under controlled oxidation of Pb-Ti thin films. Moreover, recent reviews on Pb- and Pb-Bi-based multicomponent systems underline that reliable phase equilibrium data remain limited, particularly for ternary and higher-order systems, despite their importance for predicting coolant/material compatibility in reactor environments [22].

These findings collectively suggest that both oxide stability and dissolution phenomena, as well as more complex interfacial effects, can govern the wetting behavior of Pb on refractory metals. However, the sessile drop method with a conventional contact heating procedure typically involves isothermal conditions and does not account for the dynamic thermal gradients and cyclic heating/cooling that occur in real reactor environments. Recent studies have highlighted the need for more realistic testing protocols that simulate operational conditions, including fluctuating temperatures and thermal cycling [23-26]. Such conditions can influence not only the wetting behavior but also the formation of intermetallic phases and diffusion layers at the coolant/refractory interface.

To address these challenges, the present study introduces a novel experimental methodology for characterizing the wetting behavior between a liquid metal coolant and a metallic refractory, which better mimics the real working conditions of a cooling system of a nuclear reactor. Using the proposed stepwise heating/cooling protocol combined with thermocycling, the wetting behavior of liquid lead on three key structural metals, molybdenum, tantalum, and titanium, was systematically investigated. These metals are frequently employed in advanced nuclear structural applications due to their thermal stability, corrosion resistance, and mechanical strength [1,3,6-7]. The results aim to better understanding of physicochemical compatibility at liquid metal/solid interfaces under dynamic thermal conditions, providing critical insights for material selection in LFRs and other high-temperature systems.

2. Experimental

To investigate the high-temperature interactions between liquid Pb and selected solid metallic substrates (Me = Mo, Ta, and Ti) under vacuum conditions, a custom-designed device, previously described in detail in [27], was employed. Additionally, a new methodological approach incorporated the sessile drop method combined with a stepwise heating-cooling protocol, to mimic the operating conditions of a coolant within a nuclear reactor's cooling system.. The approach was designed to enhance both measurement accuracy and reproducibility by incorporating two key elements: (1) in-situ oxide removal from the Pb drop surface using the capillary purification (CP) procedure, ensuring reliable wetting measurements, and (2) a stepwise thermal cycling program simulating reactor-like temperature variations through controlled, cyclical, and stepped temperature changes. Assuming that the maximum permissible operating temperature of the coolant in LFR-type reactors is 600°C [1,2], tests were carried out under variable thermal conditions (Fig. 1). Two experimental protocols were employed:

- Protocol I: stepwise heating from 400°C (the temperature at which Pb drops are squizeed from the capillary and deposited on the substrate) up to 600°C, with isothermal holds (~ 10 min) at each stage (400, 500, 600°C), followed by stepwise cooling to 400°C, including intermediate isothermal holds at 500°C and finally at 400°C.
- Protocol II: an additional cycle involving reheating the couple from 400°C back to 600°C, followed by cooling to 400°C, with isothermal holds at each stage. This protocol was particularly applied to the Pb/Mo and Pb/Ta couples to examine whether repeated thermal cycling affected the contact angle evolution.

This methodological framework was selected to account for the cyclic variations in temperature at the metal-liquid interface, which arise from the transport of coolant between cooler and superheated regions within the reactor [3].

The repeated heating–cooling protocol was intentionally introduced to reproduce, in a simplified and accelerated way, the thermal fluctuations occurring in operational reactor environments, where structural components are subjected to numerous thermal transients. This procedure allows for verifying whether the contact angle remains stable under repeated thermal loads or whether subtle kinetic effects (e.g., enhanced spreading, interfacial degradation, oxide reformation, or phase evolution) may appear over time. Even if no visible changes in the macroscopic drop geometry were observed, such a protocol provides a reproducible reference state and a basis for future high-resolution microstructural studies after cycling, which can capture nanoscale changes undetectable at SEM resolution.

Before high-temperature testing, substrates were mechanically polished following the established preparation procedure described in earlier studies [27,31-32]. For the Pb/Ti couples,

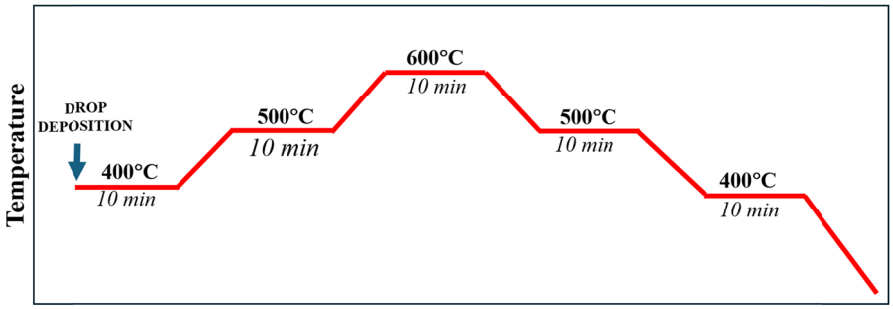


Fig. 1. Temperature profile of one stepwise heating-cooling cycle

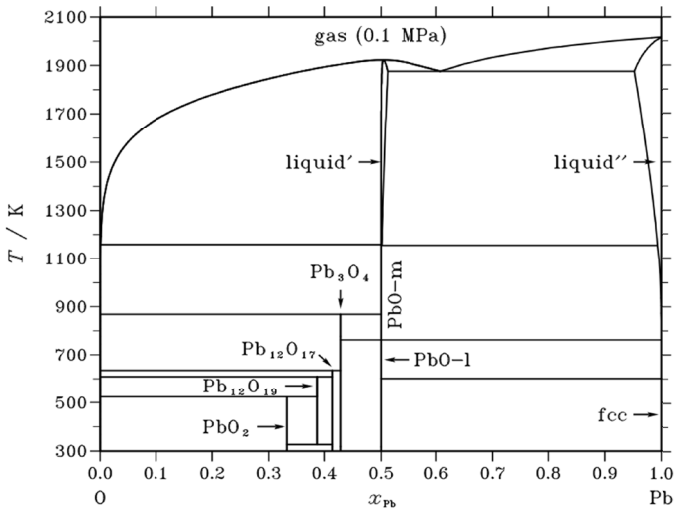


Fig. 2. The O-Pb phase diagram taken from [33]

comparative tests were conducted on substrates with two distinct surface finishes: polished (Ra = 0.3 μm) and unpolished, rough-ground (Ra = 2.0 μm). In addition, experiments were carried out using both graphite and alumina capillaries to evaluate the influence of capillary material on wetting behavior.

In order to ensure reliable wetting measurements, particular attention was paid to the removal of oxide films from the surface of the liquid lead before its contact with the substrate. Lead is known to form a variety of oxide phases (PbO, Pb_3O_4, $Pb_{12}O_{17}$, PbO_2, etc.) depending on temperature and oxygen partial pressure, as described in the O-Pb phase diagram (Fig. 2). These oxides can significantly alter the surface tension and wetting behavior of Pb, especially in the presence of oxygen impurities or during prolonged exposure to air. To eliminate this variable, the capillary purification (CP) procedure [26-31] was employed. This method allows for in-situ deposition of Pb drops from a capillary

under vacuum conditions, effectively disrupting and removing the native oxide film. The necessity of this approach is supported by thermodynamic modeling of the O-Pb system, which predicts the formation of stable oxide phases even at relatively low oxygen activities. Without purification, the presence of PbO or PbO_2 at the interface could lead to misleading contact angle values and hide the true wetting behavior of Pb on refractory substrates. Furthermore, the CP procedure ensures that the drop surface is freshly exposed and free from contamination, enabling reproducible and interpretable wetting measurements. This is particularly important given the sensitivity of Pb to oxygen uptake and the tendency of oxide formation to influence interfacial phenomena, as observed in other studies involving Pb-metal systems.

Just before the experiments, the surface of the Pb shots (99.99 wt.%) was mechanically polished with SiC papers and then ultrasonically cleaned in isopropanol for approximately 15 minutes. The cleaned and degreased Pb sample was placed in a graphite or alumina capillary and immediately transferred to the experimental chamber. Subsequently, the substrates of selected pure metals (Me = Mo, Ta, Ti, purity ≥99 wt.%) were located on holders, and the measurement chamber was evacuated with a set of pumps to a pressure of 10^{-6} mbar. The flushing procedure was then carried out twice by introducing inert gas (pure Ar, 99.9999 wt.%) to a pressure of 1.10×10^{3} mbar, which was then pumped off again. After the last flushing, when the vacuum once again reached 10^{-6} mbar, non-contact heating of the capillary with the Pb sample was started to about 150°C to purify the atmosphere by continuous vacuuming and removing adsorbed residual water vapor and other volatiles from the surfaces of the capillary and other parts of the measuring chamber. Finally, when the vacuum achieved 10^{-6} mbar, the device was heated to the first start temperature of 400°C, and then the selected substrate was introduced into the experimental chamber.

After sessile drop tests, the solidified Pb/Me couples were subjected to detailed microstructural characterization using scanning electron microscopy (SEM) and back-scattered detector (BSE), combined with energy-dispersive X-ray spectroscopy (EDS), to evaluate the morphology and composition of the interfacial zones.

3. Results and discussion

The stepwise thermal cycle procedure applied in this study enabled a detailed evaluation of the wetting behavior of liquid Pb on refractory metals under conditions representative of a nuclear reactor's operation. Despite the differences in chemical reactivity and phase equilibria of Mo, Ta, and Ti with liquid Pb, several general conclusions can be drawn.

3.1. Wetting tests

The wetting behavior of liquid Pb on Mo, Ta, and Ti substrates was systematically investigated using the stepwise thermal cycle protocol. The experiments were monitored simultaneously by two cameras positioned perpendicularly to each other: one in shadow mode for contact angle determination, and the other in real-time view for in situ recording of the evolution of the Pb drop surface. Importantly, during the entire long-term experiments (~ 3600-5400 s), including multiple heating-cooling cycles, the Pb surface remained bright and shiny, with no signs of surface matting or particle formation, as shown in the example of the Pb/Mo couple in Fig. 3. This observation provides strong evidence of both the high purity of the vacuum atmosphere and the effectiveness of the CP procedure in removing native oxide films from the Pb drop before its deposition on the substrate surface. Such control of the atmosphere and oxide removal is essential, as earlier studies have shown that surface oxides strongly affect measured contact angles and kinetics of wetting [12-21,27-32,34,35].

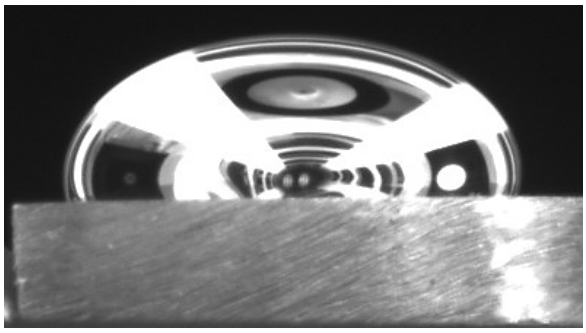


Fig. 3. Image recorded by a CCD camera in real-time view during the study of wetting behaviour of Pb/Mo couple in a vacuum; the image shows Pb/Mo couple after a stepwise thermal cycle at a final stage of 400°C

In the case of Pb/Mo couples, as shown in the wetting kinetics in Fig. 4, liquid Pb does not wet the Mo substrate during the entire two-stepwise thermal cycles (protocol II). Immediately after deposition of the Pb drop on the Mo substrate (t = 0 s), the first contact angle formed at 400°C was $\theta_{0\_Pb/Mo}$ ~ 115°. The sudden decrease in the contact angle observed after about 150 seconds is due to the unintentional addition of an extra portion of liquid metal (see video, QR code). After that, the obtained contact angle value ~99° remained unchanged during the heating and cooling cycle of 400-600-400°C, additional reheating to 600°C, and final cooling to 400°C.

For the Pb/Ta couple, a similar initial high contact angle value ($\theta_{0\_Pb/Ta}$ ~ 118°) was observed at 400°C, followed by a gradual decrease to $\theta_{f\_Pb/Ta}$ ~ 87° upon heating to 600°C. This indicates a non-wetting to wetting transition. The subsequent cooling and reheating steps did not significantly affect the final contact angle. In this case, the observed gradual spreading can be explained by temperature-controlled spreading, in which the decrease in surface and interfacial tension with increasing temperature promotes spreading in the absence of chemical reactivity [34,35]. This highlights the importance of distinguishing between reactive and purely physicochemical wetting mechanisms. Nevertheless, the possibility of a nanometer-scale oxygen solution in Ta cannot be excluded, and a strong affinity of Pb for

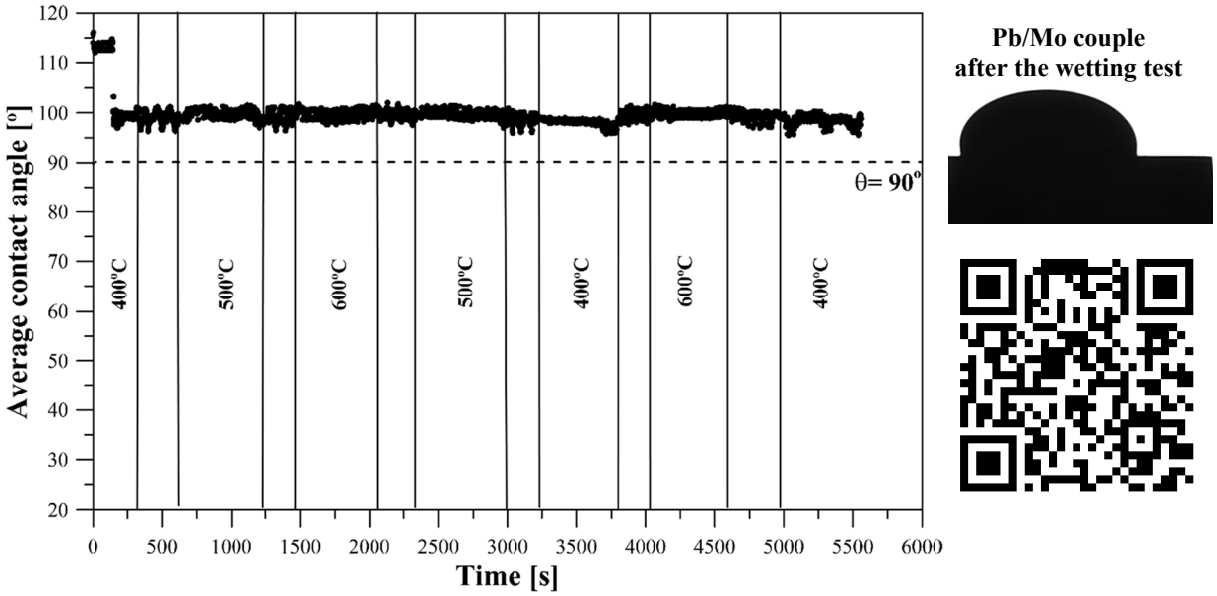


Fig. 4. Wetting kinetics of liquid Pb on Mo substrate captured during the sessile drop test with the CP procedure and two-stepwise thermal cycles (protocol II) under high vacuum, together with side-view drop profile recorded by the CCD camera in shadow mode during the high-temperature measurements at the end of the test, and QR code for the real-time video of the experiment

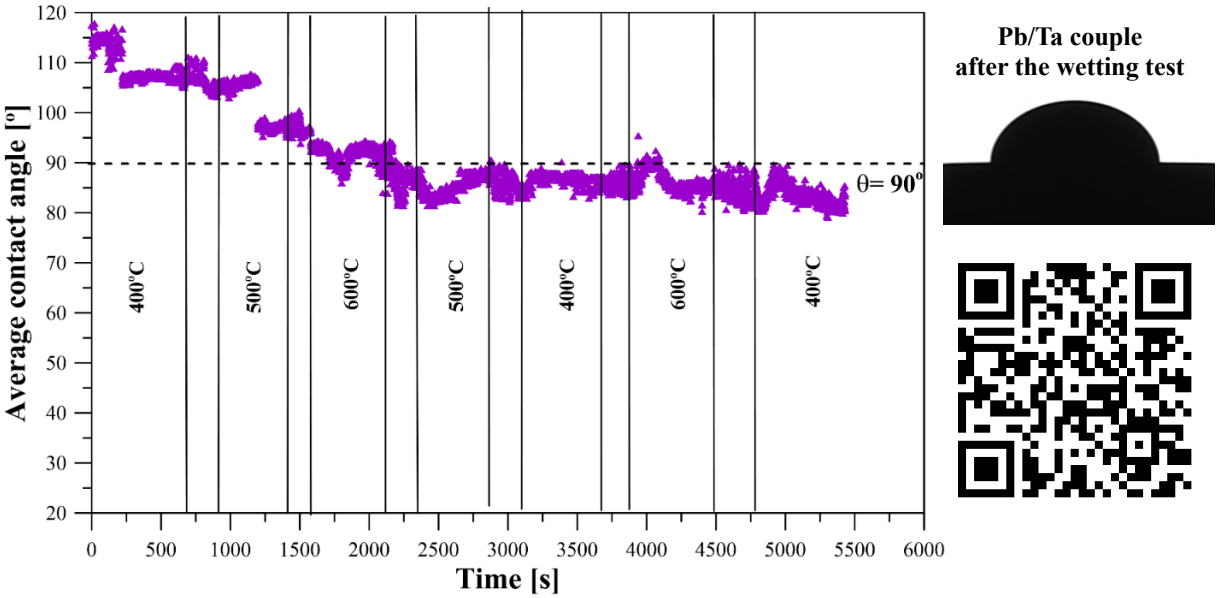


Fig. 5. Wetting kinetics of liquid Pb on Ta substrate captured during the sessile drop test with the CP procedure and two-stepwise thermal cycles (protocol II) under high vacuum, together with side-view drop profile recorded by the CCD camera in shadow mode during the high-temperature measurements at the end of the test, and QR code for the real-time video of the experiment

oxygen may facilitate subtle, submicroscopic redistribution at the interface. Resolving these effects requires future high-resolution structural characterization of surfaces and interfaces in the Pb/Ta couple.

The Pb/Ti couple displayed the most pronounced changes. The initial contact angle values are quite similar and varied between 105° and 110°, depending on substrate surface finish (polished vs unpolished) and the type of capillary material used (graphite vs alumina).

For the polished substrates (Ra = 0.3 μm) used in the tests with graphite and alumina capillaries (Figs. 6 and 7), a rapid decrease in contact angle was observed either during the isothermal hold at 500°C or during heating to 600°C. In the former case, the final contact angle reached ~30°, while in the latter, it stabilized at ~67°. Despite the difference in absolute values, both cases indicate strong interfacial interaction and pronounced wetting behavior. The observed differences may be because graphite may partially react with lead oxide at elevated temperatures, acting as an effective oxygen getter [36,37]. This can reduce the local oxygen activity, thereby enhancing the chemical purity of the liquid lead and promoting stronger interfacial reactivity, most notably, by the dissolution of Ti into the liquid Pb. For the conditions used, alumina is chemically inert, and it probably did not provide such a reducing effect. These findings emphasize that,

beyond substrate preparation, capillary material and purification procedures critically might influence the measured wetting kinetics in liquid Pb/solid couples.

On an unpolished (Ra = 2.0 μm), rough-ground Ti substrate (Fig. 8), the liquid Pb drop initially exhibited a contact angle similar to that observed on a polished surface. No significant changes were observed during the isothermal hold at 400°C and the early stage of heating at 500°C. However, the contact angle decreased sharply to ~65° midway through the 500°C step, and further reduced to 28° during the 600°C stage. This last value remained stable throughout the subsequent cooling phase down to 400°C. It is worth noting that the observed difference in contact angle on the rough-ground surface confirmed the well-known influence of surface roughness on apparent wettability [28,30,34]. It should be highlighted that in this case, the contact angle was measured in a plane parallel to the grinding direction, which led to an asymmetric drop shape due to preferential drop spreading along the grooves formed during unidirectional grinding of the substrate.

3.2. Microstructure observations

Cross-sectional SEM/BSE observations combined with EDS elemental mapping were performed to assess the microstructural evolution at the Pb/Me interfaces. As an example,

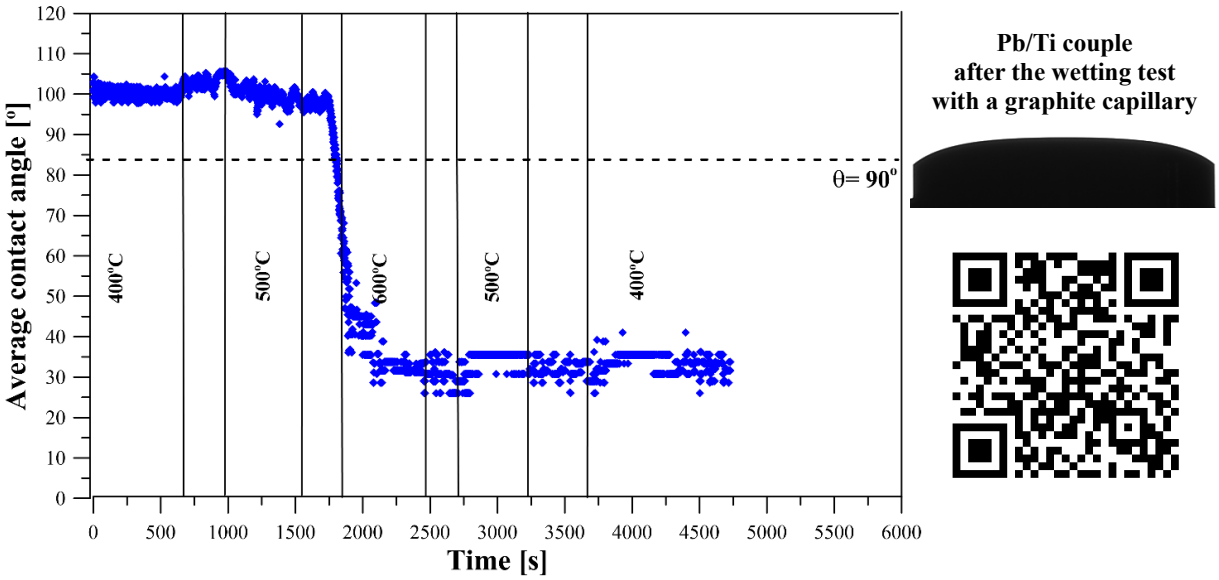


Fig. 6. Wetting kinetics of liquid Pb on polished Ti substrate obtained with the use of graphite capillary and captured during the sessile drop test with the CP and stepwise thermal cycle procedure (protocol I) under high vacuum, together with side-view drop profile recorded by the CCD camera in shadow mode during the high-temperature measurements at the end of the test, and QR code for the real-time video of the experiment

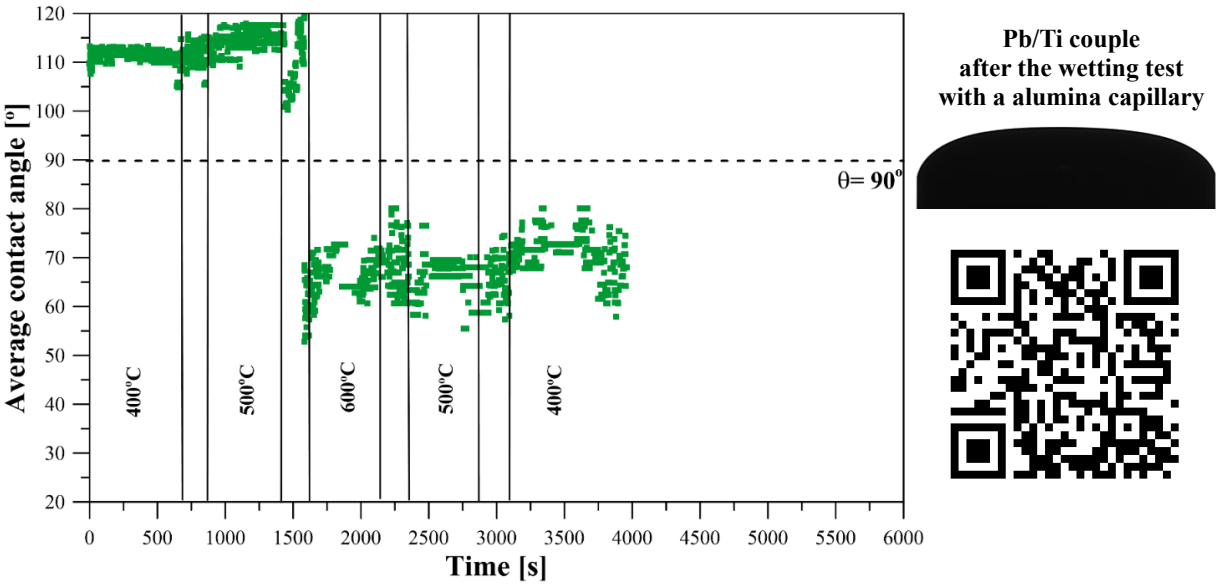


Fig. 7. Wetting kinetics of liquid Pb on polished Ti substrate obtained with the use of alumina capillary and captured during the sessile drop test with the CP and stepwise thermal cycle procedure (protocol I) under high vacuum, together with side-view drop profile recorded by the CCD camera in shadow mode during the high-temperature measurements at the end of the test, and QR code for the real-time video of the experiment

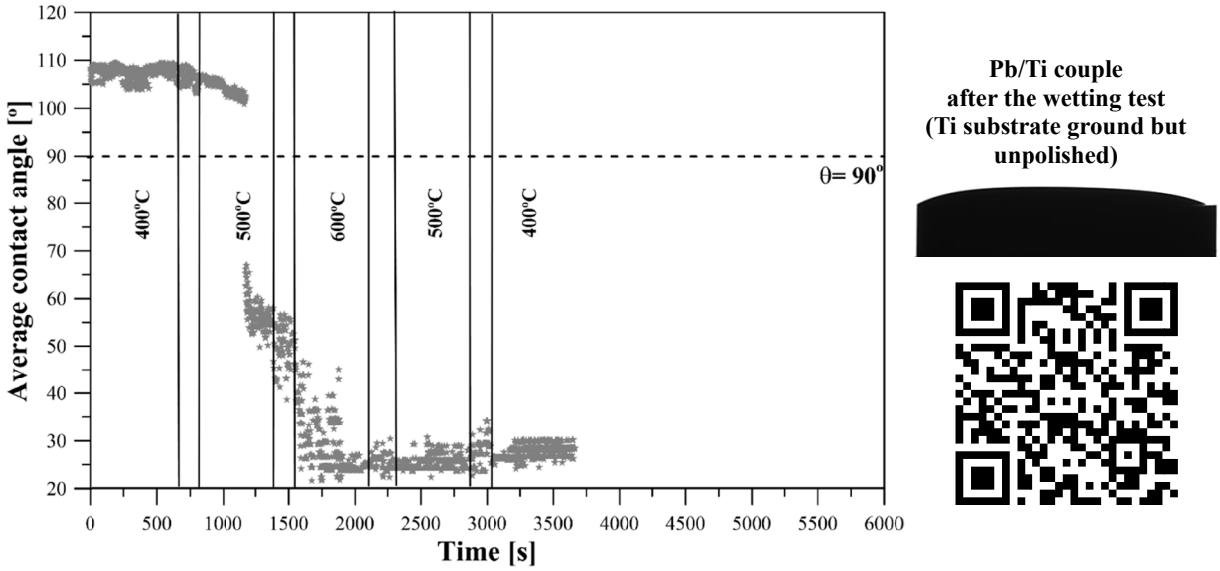


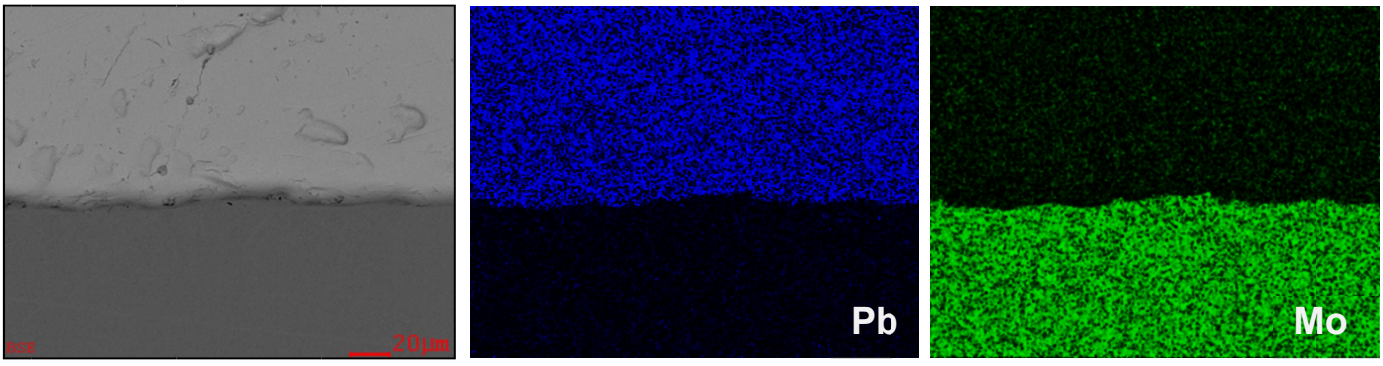
Fig. 8. Wetting kinetics of liquid Pb on unpolished Ti substrate obtained with the use of alumina capillary and captured during the sessile drop test with the CP and stepwise thermal cycle procedure (protocol I) under high vacuum, together with side-view drop profile recorded by the CCD camera in shadow mode during the high-temperature measurements at the end of the test, and QR code for the real-time video of the experiment

results for the Pb/Mo couple (Fig. 9) were presented, since the observations for Pb/Ta and Pb/Ti were qualitatively similar under the magnifications employed in this study.

In the Pb/Mo system, no evidence of interfacial reaction layers or compound formation was detected within the resolution and magnification used in this study. The interface appeared quite sharp, with the Pb drop in direct contact with the Mo substrate. These results confirm the predictions from the Mo-Pb phase diagram, which show negligible mutual solubility and no intermetallic compound formation [38]. This observation agrees with earlier scanning tunneling microscopy investigations by [20] who reported that ultrathin Pb films on Mo(110) exhibit non-trivial quantum size effects, forming stable islands with specific layer heights rather than uniform films. While such nanoscale ordering phenomena are unlikely to influence macroscopic sessile drop wetting, they emphasize that Pb/Mo interfaces are governed by subtle electronic interactions even in the absence of bulk reactivity. Thus, the limited wetting observed in this study may reflect the intrinsic inertness of the Pb/Mo system under reactor-relevant thermal conditions.

It is important to emphasize that the absence of observable interfacial products does not exclude possible interactions at the nanoscale. The preparation of cross-sections was particularly challenging due to the significant differences in hardness and plasticity between Pb and refractory substrates, often leading to Pb smearing during polishing and potential masking of subtle interfacial features. Moreover, Pb is highly ductile, which complicates reliable mechanical sectioning and can obscure very thin reaction zones. For this reason, the present results should be considered as first-of-their-kind, baseline observations, requiring further high-resolution structural studies. Future investigations employing advanced techniques such as transmission electron microscopy or high-resolution scanning electron microscopy will be essential to reveal whether nanoscale dissolution, segregation, or intermetallic phase formation occurs at the Pb/refractory metal interfaces.

In addition, the interpretation of SEM/EDS results is further complicated by the lack of reliable phase diagram data for the Pb-Ta and Pb-Ti systems, particularly on the Pb-rich side. For Pb-Ta, no experimentally validated phase equilibria are currently



(a) (b) (c)

Fig. 9 SEM/BSE image (a) of the cross-sectioned Pb/Mo couple at a magnification of 1000×, together with the related EDS maps of elements (b,c)

available, while for Pb-Ti, only fragmentary information exists, limited to the Ti-rich region [39]. This absence of thermodynamic reference data prevents unambiguous conclusions regarding whether the apparent absence of reaction products in SEM is due to true non-reactivity, or whether extremely thin layers or metastable phases could have formed but remained below the detection threshold. Consequently, while our findings strongly suggest limited or non-reactive wetting behavior at the resolution employed, the results must be viewed in the context of these fundamental knowledge gaps, further reinforcing the need for both advanced structural characterization and thermodynamic modeling in future work.

3.3. Significance of the stepwise thermal cycle procedure

A key insight from all experiments is that temperature influences wetting behavior primarily during the heating phase. While heating to and/or holding at the maximum temperature (600°C), the contact angle stabilization occurs and persists during the subsequent cooling. This observation suggests that reactions at the liquid/solid interface are thermally activated and possibly irreversible within the test timescale.

This approach also enabled continuous monitoring of the Pb drop surface and interface stability under dynamic conditions. The fact that the Pb drop remained bright and unaltered throughout all tests strongly indicates both the effectiveness of oxide removal and the high purity of the testing atmosphere. In contrast to many previous studies [12-21], our results confirm that the stepwise thermal cycle protocol combined with a capillary purification procedure allows for reliable and reproducible characterization of wetting kinetics. Moreover, it provides a more representative simulation of a nuclear reactor's operating conditions and offers a reproducible method to study the transient response of structural materials to high-temperature liquid metal exposure.

4. Conclusions

This study demonstrates the effectiveness of a newly developed stepped heating–cooling protocol combined with the capillary purification procedure for investigating the physicochemical interaction between solid metals and liquid lead used as a coolant in nuclear reactor structures. The following conclusions can be drawn:

1. The wettability of Pb on Mo, Ta, and Ti is significantly influenced by temperature, but only during the stepped heating phase, while contact angles remain stable during subsequent cooling and additional heating cycle.
2. Mo and Ta exhibit poor wettability (depending on temperature, $\theta_{Pb/Mo}$ = 115-100° and $\theta_{Pb/Ta}$ = 87-84°, respectively) and no evidence of chemical reactivity with Pb, suggesting their suitability in applications requiring chemical inertness.
3. The Pb/Ti system exhibited the wetting behavior among all couples studied, with contact angles decreasing from ~105-110° to values as low as 28-30° depending on substrate surface finishing and capillary type. The final contact angle value registered for polished Ti and graphite capillary was $\theta \sim 30°$, for polished Ti and alumina capillary it was $\theta \sim 67°$, while for unpolished Ti and alumina capillary it was $\theta \sim 28°$. Overall, these results indicate that both interfacial chemistry and experimental parameters critically govern the wetting behavior of Pb on Ti.
4. The stepwise thermal cycle protocol allows for a more realistic simulation of reactor conditions, as compared to conventional isothermal tests, providing valuable insights for the selection and design of structural materials in lead-cooled systems.

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