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EXPRESS ARTICLE

Improvements in experimental investigation of molten Mg-based materials

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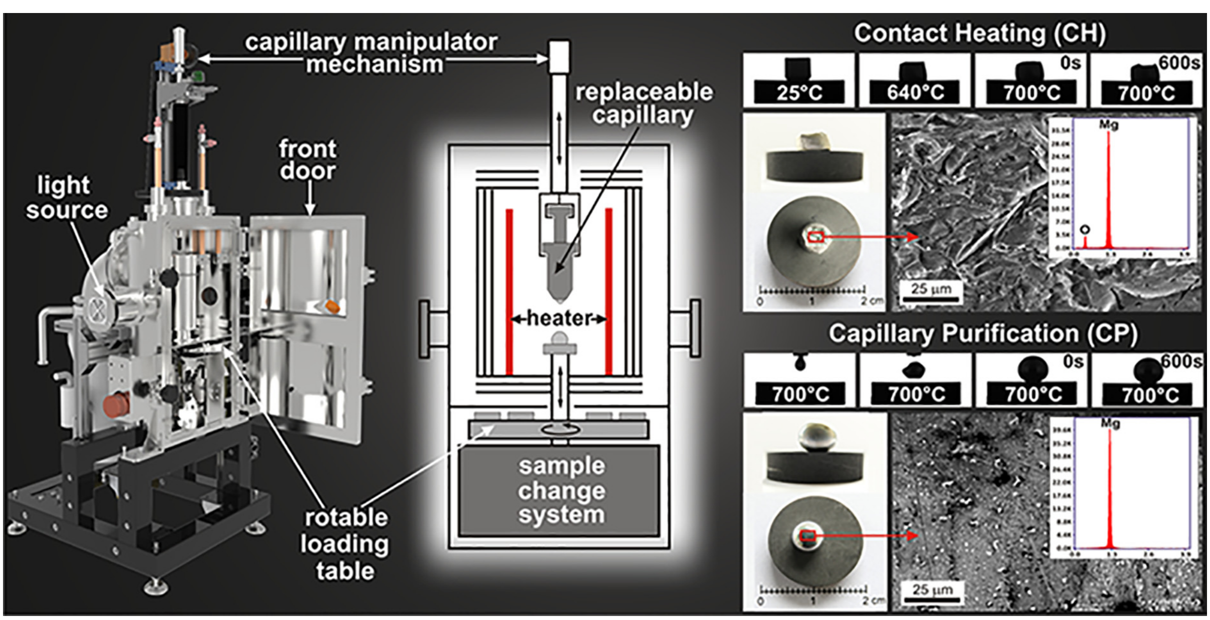
ABSTRACT

In this paper, the experimental performance of a new testing device designed for investigating the high temperature properties of molten Mg is presented. The newly developed device allows examining high temperature wetting behavior and thermophysical properties of molten Mg (and Mg alloys) by using various experimental procedures (e.g. classical sessile drop, pendant drop, dispensed drop and drop sucking). High temperature wettability tests at temperatures up to 1000 °C in an inert gas atmosphere or under high vacuum (up to 10^{-7} hPa) are now possible. It has been documented that the application of the classical sessile drop method combined with a capillary purification procedure successfully eliminates the problem of magnesium oxidation that traditionally affects obtained results. Selected examples of high temperature experiments carried out for molten Mg in contact with various refractories are presented in order to show a wide range of analytical possibilities of the new device. The results obtained by using the new device are important from both a high theoretical and practical perspective regarding liquid phase assisted fabrication and processing of Mg-based alloys and metal-matrix composites.



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High-strength and lightweight Mg-based metal matrix composites (MMCs) exhibit competitive advantages in aerospace, automotive, military and medical applications [1]. Among various fabrication methods of Mg-based MMCs, liquid-phase assisted technologies (e.g. stir casting or squeeze casting) provide relatively low costs, high efficiency and process simplicity [2]. However, in order to ensure a high quality of fabricated Mg-based composites, it is necessary to investigate all the interfacial phenomena taking place between molten Mg and several ceramics as reinforcement candidates that include thermodynamics, thermophysical properties and wetting characteristics.

Sessile drop (SD) and large drop (LD) methods are commonly applied to measure a set of thermophysical properties of molten metals (e.g. surface tension or density) [3]. Additionally, the SD method affords the ability to determine wettability characteristics of metal/metal or metal/ceramic systems such as contact angle (θ), spreading kinetics $\theta = f(t)$ and the effects of adhesion. On the other hand, experimental investigations performed on molten Mg by the SD method are very challenging, owing to the well-known pronounced oxidation and evaporation tendencies of Mg at the liquid state negatively affecting the reliability of the results obtained. In addition, due to a very intensive evaporation of Mg when obtaining high temperature measurements, complex and expensive components of experimental devices are subject to being severely contaminated or even seriously damaged.

Due to the aforementioned reasons, few studies are reported on the wettability of Mg-based materials with refractories. A literature survey established that most of the available papers (e.g. [4]) report experiments performed using the “classical” contact heating SD method procedure (CH). However, this procedure does not allow purifying the surface of molten metal from a native oxide layer negatively affecting the quality of obtained results as stated earlier.

The effect of evaporation and oxidation of Mg during wettability tests has been described in detail by [5–7]. Contreras et al. [6] which proved a strong effect of the oxide film segregated at the Mg droplet surface on the spreading kinetics measured by the SD/CH procedure. In order to obtain “true” contact angle values, the testing temperature was increased to 950 °C (300 °C above the melting point of Mg). Consequently, at such high temperatures the surface oxide film was thermally removed and a true contact angle at the Mg/TiC triple line was measured. However, carrying out wettability tests at temperatures much higher than the melting point of Mg results in a substantial overheating and evaporation of the material. Moreover, a massive contamination of the test chamber could result significantly affecting the phase equilibria and chemical reactions taking place during any subsequent high temperature tests. Thus, a strong need for receiving valuable data on the behavior of molten Mg as well as the limitations of existing test apparatuses provided the impetus to develop an experimental device having improved design concepts to address these challenges [8]. Although testing apparatus previously developed [9] shows unique experimental performance, mainly in meeting all the requirements to properly investigate liquid metals and metal/ceramic interfaces at high temperature, the new ad-hoc device developed for investigating liquid Mg and Mg-based alloys includes additional features: (i) molybdenum as a heating element to reduce the residual oxygen content inside the chamber; (ii) a simplified set-up of the experimental chamber allowing easy-access and fast cleaning; (iii) the ability to place samples onto a rotating table to both quench the sample (to contain evaporation) and to provide the ability to examine more than one sample (up to 10) under the same test conditions (atmosphere, temperature etc.) during the same experiment; (iv) the availability of various testing methods and procedures (e.g. SD/LD, pendant drop, dispensed drop, drop sucking) with a particular emphasis on the capillary purification procedure (CP); (v) shutters to protect viewports; (vi) protective screens for sensors and filters for the pumps; (vii) a possibility to perform experiments under a vacuum or under a static/flowing atmosphere of inert gas; (viii) gas analysers and (ix) the high speed recording of drop images (up to 2000 frames/s) to enable a real-time analyses of the experiment(s).

The main advantage of the CP procedure is the purification of the droplet surface from its native oxide layer mechanically removed by squeezing the molten Mg through a hole in capillary. Furthermore, the presence of Mo as the heating element (acting as an O-getter), combined with an imposed atmosphere surrounding the molten metal suppresses further oxidation/adsorption phenomena from occurring at the surface.

In order to test its experimental performance and the applied testing procedure on the reliability of the obtained results, wetting experiments were performed by simultaneously using CH and CP procedures. The materials investigated were pure Mg (99.98%) (Stanchem, Poland) and mechanically polished (surface roughness ~ 150 nm) graphite as a substrate (Kryzaplast, Poland). In order to limit evaporation, wetting tests were carried out under an atmosphere of flowing Ar (99.999%). The outcomes confirmed that in the case of a CH procedure the presence of a native surface oxide layer (evidenced by the high oxygen content detected by SEM/EDS analysis) hindered the complete melting of the Mg. Consequently, a real Mg/C interface was not formed and the irregular geometry of the partially molten drop did not permit an analysis of the spreading kinetics. On the contrary, a different behavior of the molten Mg was observed during the test performed using a CP procedure. A complete molten drop was squeezed through a C-capillary and deposited onto graphite at $T = 700$ °C and kept under the same operating conditions for 600 s. Subsequently, during the whole test period the regular shape of drop did not change, allowing the measurement of the contact angle values and spreading kinetics. The results of the CP experiment indicate that the Mg/C system exhibits a non-wetting behavior (measured by a contact angle of $\theta \sim 150°$). Moreover, the top of the solidified Mg drop was perfectly smooth having a mirror-like surface, typical for a non-oxidized metal surface. The absence of oxides and Mg-carbides at the surface was confirmed by SEM/EDS analyses performed at the top of Mg-solidified drop. At $T = 700$ °C, the same $\theta = f(t)$ behaviour is reported in [5] where the non-reactive Mg/MgO behaviour as a function of temperature was investigated.

In summary, the new concept apparatus presented here provides a clear advancement for examining the high temperature behaviour of molten Mg potentially offering flexibility and a wide spread of capabilities in experimental design. Indeed, the experiments might be performed under well-defined operating conditions by using different testing methods and procedures. We expect that experimental outcomes obtained by using this equipment will provide a valuable input for both theoretical and experimental research on liquid-phase assisted testing and processing of Mg-based alloys and composites.

Author contribution section

AK, NS and JB have participated in designing and construction of the high temperature device, while WP and DG have participated in high temperature sessile drop experiments and overall preparation of this manuscript.

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