Experimental investigation on in-situ microwave casting of copper

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Abstract. The in-situ microwave casting of metallic materials is a recently developed casting process. The process works on the principles of hybrid microwave heating and is accomplished inside the applicator cavity. The process involves – melting of the charge, in-situ pouring and solidification of the melt. The electromagnetic and thermal properties of the charge affects microwave-material interaction and hence melting of the charge. On the other hand, cooling conditions inside the applicator controls solidification process. The present work reports on in-situ casting of copper developed inside a multimode cavity at 2.45 GHz using 1400 W. The molten metal was allowed to get poured in-situ inside a graphite mold and solidification was carried out in the same mold inside the applicator cavity. The interaction of microwave with the charge during exposure was studied and the role of oxide layer during melting the copper blocks has been presented. The developed in-situ cast was characterized to access the cast quality. Microstructural study revealed the homogeneous and dense structure of the cast. The X-ray diffraction pattern indicated presence of copper in different orientations with (1 1 1) as the dominant orientation. The average micro indentation hardness of the casts was found 93±20 HV.

1. Introduction
Processing of metallic materials using microwave energy was considered an impossible task owing to reflection of microwaves from metallic surfaces [1]. Roy et al. demonstrated that metallic powder can be processed using microwave energy [2]. Metallic materials in powder form couple with microwaves due to their higher surface area and it results in heat dissipation inside the target materials [3]. Use of microwave hybrid heating (MHH) further enhances the heating uniformity [4] and saves energy with compression in processing time [5, 6]. These attributes of MHH were exploited during sintering [7-10], joining [11, 12], cladding [13, 14] and melting [15-17] of metallic materials using microwave energy. Improvement in the properties of microwave processed parts were reported using these advanced processes while compared to the conventional processing techniques. The rapid and selective heating characteristics of microwave energy were explored during joining and cladding processes and better properties of the microwave processed joints and clads were demonstrated while compared to the conventional processes.

Few researchers demonstrated that use of microwave energy for melting of metallic materials significantly reduces the energy consumption, processing time [15, 17] and offers an eco-friendly
route as compared to conventional heating techniques. In spite of these interesting results, microwave melting was not exploited adequately for casting applications. Microwave casting of metallic materials was explored by a few researchers in the recent years [18-24]. Microwave casting of Ni-based powder matrix SiC composite was reported with better distribution of reinforcement with equiaxed grains [18]. Further, microwave casting of bulk metals were also reported. In a study, conventional casting of the bulk Al alloy 1050 was reported [21]. Improvement in mechanical properties of the microwave cast was observed as compared to the as-received alloy. In another approach, in-situ microwave casting of the lead [3], copper [3] and aluminium alloy 7039 [19] was reported; the principals involved in the process and processing method were also discussed [19]. Finer microstructure and improved mechanical properties were observed in the developed in-situ casts of AA 7039 [23]. Techniques to improve the grain structure and influence of grain and intermetallic precipitates on indentation hardness during these techniques were also explored [22, 24]. Theoretical study of the process suggested that use of higher power and suitable location for charge to be placed inside the applicator cavity improves melting time [20]. It was reported that microwave interaction with different bulk metallic materials for its melting during the in-situ microwave casting depends upon its electromagnetic and thermal properties [1, 15]. Melting of AA 7039 in an ambient atmosphere was analysed during the in-situ casting process which revealed that oxide layer plays an important role during in-situ microwave casting process [19]. Microwave heating characteristics of copper was also reported in an argon environment [15]; however, such no data were reported in the ambient atmosphere which is more important to understand the effect of CuO during heating of copper for efficient melting. It is, therefore, the objective of the present study was to explore the in-situ microwave casting of copper. The casts were developed at 2.45 GHz and 1400 W. Influence of oxide layer during melting of the copper was analysed. The developed in-situ copper casts were characterized to study the microstructure and indentation hardness properties.

2. Experimental

Schematic diagram of the setup used for the in-situ casting process using microwave energy is shown in figure 1. An industrial microwave applicator (Model: MH-1514-101-V6, Make: Enerzi Microwave Systems Pvt. Ltd.) was used to irradiate the charge. A mold assembly was placed inside the cavity to develop the copper castings. The cavity was water cooled and a built-in pyrometer (infrared, range: 350 °C–1800 °C and least count: 1 °C) was provided for temperature measurement of the target material. Commercially available pure copper (99.95 wt%) was used as the target charge during the casting trials (figure 1, inset). A ceramic crucible, which absorbs microwaves rapidly, was used for hybrid heating of the charge which also acts as the pouring basin. A graphite split mold, consisting of cope and drag, was used to cast the molten alloy in-situ inside the mold cavity. Silicon Carbide (SiC) plates were used as the susceptor to enhance the heating rate during the MHH of the charge. The refractory insulation was used to cover the mold assembly inside the applicator during irradiation to prevent heat losses from the mold assembly. An alumina base was used to place the SiC and mold to avoid chemical reaction between SiC and insulation block at elevated temperatures as shown in figure 1. The charge was placed inside the pouring basing and microwave exposure was carried out. The system was exposed to microwaves for approximately 890±10 s at 1400 W and the temperature of the top surface of charge was monitored using the IR pyrometer. A sharp dip in the monitored temperature confirms the in-situ pouring of the molten copper inside the mold cavity. The molten metal was then allowed to solidify inside the mold cavity; a typical cast developed is shown in figure 1 (inset). The test specimens for characterisation were prepared from the casts and standard metallographic techniques were used to polish them. A scanning electron microscope (SEM; Model: LEO 435VP) was used to analyse the microstructure of the cast. An x-ray diffractometer (Model: D-8 Bruker AXS diffractometer with Cu-Kα) was used to study the phases of the cast using the scan rate of 1° min⁻¹ within the range 5°-100°.
3. Results and discussion

3.1. Heating of the charge

The role of oxide layer that was formed during the microwave heating of the bulk copper is schematically presented in figure 2. It was reported that the bulk copper contains two layers of oxides at its surfaces in contact at the ambient atmosphere [25]. Generally, Cu$_2$O (2 nm) first grow on the outer surface of the bulk copper and then a thin layer of CuO (1.3 nm) is formed on the Cu$_2$O layer as shown in figure 2 (a) at room temperature ($T_R = T_M$, metal temperature). The oxide CuO is an excellent absorber of microwave energy and its dielectric properties enhances significantly with increase in temperature [26]. As the microwave exposure starts, these layers of oxides start microwave absorption (Cu$_2$O absorbs at a relatively elevated temperature, though); however, microwave coupling with the bulk copper depends upon its critical temperature ($T_C$). The $T_C$ of a bulk metal is a temperature beyond which it absorbs microwave energy dominantly. Thus, the bulk copper gets heated through conventional modes of heating through susceptor, pouring basin and oxide layers as shown in figure 2b.

![Figure 2. Schematic of different phases involved in microwave heating of the bulk copper](image)

The microwaves which reach to the surface of copper get reflected due to very small skin depth of the metal below its critical temperature. The oxide layer thickness increases as the temperature of the copper increases and microwave absorption in oxide layer also improves. The absorption of microwave energy in the bulk copper starts as its temperature reaches beyond its critical temperature.
(\(T_c\)) due to conventional heating. The heating of the copper beyond this phase takes place due to the MHH. At elevated temperature, the oxide layer thickness further increases due to enhanced reaction of copper ions with ambient air. A scanning electron image of the oxide layer formed during melting of the copper charge is shown in figure3a. The energy dispersive spectroscopy (EDS) results of the as-received and oxide layer (taken on the same material shown in figure 3a) is presented in figure 3b. It was revealed by the EDS results that presence of oxygen got enhanced significantly in the oxide layer as compared to the as-received copper at the end of microwave exposure. It indicates possible presence of copper oxides (CuO and Cu$_2$O) in the oxide layer which enhanced microwave energy absorption during the hybrid heating. Thus, microwave absorption in copper gets increased which results in rapid and more uniform heating of the charge as shown in the figure2c.

**Figure 3.** Typical (a) scanning electron image of oxide layer obtained after in-situ pouring and (b) EDS results of as-received and oxide layer.

3.2. Characterization of the cast

The XRD spectra of the two typical casts (Cast 1 & Cast 2) are shown in figure4. It is revealed by the spectra that the presence of copper peaks of the FCC lattice are dominant with trace of Cu$_2$O and CuO. Major peak of Cu (at \(2\theta = 43.49^\circ\)) with (1 1 1) orientation indicates maximum intensity; whereas, other minor peaks with (2 0 0), (2 2 0), (3 1 1) and (2 2 2) get significantly attenuated.

**Figure 4.** Typical X-ray diffraction spectra of the cast

Typical SEM images of the in-situ cast are shown in figure5. The images clearly indicate that the copper cast is homogeneous and dense. A few casting defects, for example, inclusions (figure 5a) and micropores (figure 5b) could be identified. The porosity of the cast was found to be approximately 2–
5%. The observed homogeneity of the cast is attributed to the preheating of the mold inside the applicator cavity as the mold material graphite itself is a microwave susceptor and therefore gets heated faster than the metal. Further, the microwave applicator cavity was designed to maintain a safe temperature through water circulation around it; consequently, the enhanced cooling condition of the cavity did influence the solidification process in developing finer and more homogeneous grains of the casts.

![Figure 5](image-url) Typical SEM images of the sections of in-situ cast

The micro indentation hardness of the in-situ copper casts was accessed using a Vicker's micro indentation hardness tester (Make: Chennai Metco). The load of 100 g was applied for 30 s to develop the indentations. The average micro indentation hardness of the in-situ casts was observed to be 93±20 HV. The micro indentation hardness obtained in the present study is comparable with the previously reported micro indentation hardness (80 HV [27] and approximately 100 HV [28]).

4. Conclusions
In-situ microwave cast of the bulk copper was developed using MHH at 2.45 GHz in a graphite mold. The process requires a suitable susceptor to initiate heating and raising the charge temperature beyond the material specific critical temperature. The oxide layer plays a significant role in heating the bulk copper during MHH. The homogeneous and dense structure of the in-situ cast reveals potential of the process. The observed porosity of the in-situ casts (2-5%) could be a concern. The average micro indentation hardness of the casts was observed to be 93±20 HV.

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