Realization of Fe–C Eutectic Point Using an Alumina Crucible

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Abstract As another crucible material for metal–carbon eutectic points, alumina ceramic was used in the first trial to make an Fe–C eutectic point for the calibration of a thermocouple. Its melting and freezing behavior was tested 26 times with a type S thermocouple at various melting offset temperatures, namely, +4 °C, +9 °C, and +19 °C, and at a fixed freezing offset temperature of −11 °C. The melting emf is reproducible independent of the melting offset temperatures, and the standard deviation of the 26 melting temperatures is 0.02 °C without breakage of the cell. The difference of melting emf between alumina Fe–C and graphite Fe–C fixed points is only 25 mK within an uncertainty of 0.39 °C (k = 2). The melting behaviors of an alumina cell are quite similar to a common graphite cell. Thus, alumina can be used as a crucible material in an Fe–C eutectic system without breakage, and it can be used at a higher temperature range. As possible application systems using alumina crucibles, Pd–C and Si–SiC eutectic points are suggested.

Keywords Alumina crucible · Fe–C eutectic point · Melting temperature · Reproducibility · Thermocouple

1 Introduction

Metal–carbon (M–C) eutectic points are very promising as secondary fixed points at temperatures higher than the Cu freezing point. The melting temperatures of some selected M–C eutectic points are expected to be assigned by CCT WG2 (Working Group) in cooperation with WG5. These points can be used to calibrate thermometers...
or to check the linearity of a radiation thermometer. Particularly Fe–C, Co–C, Pd–C, and Pt–C eutectic points have been widely investigated for the calibration of thermocouples [1–6]. In addition, international comparisons of the M–C points by using thermocouples as transfer artifacts are in progress at EUROMET and the APMP [7].

For these important M–C cells, graphite is always used as a crucible material because it is one of the constituent elements of eutectic alloys and is inert chemically. In spite of the absence of reporting on the compositional change by successive melting and freezing reactions in the M–C system, the feasibility of the composition inside the alloy being changed to a hypereutectic composition is considered reasonable due to the excess of carbon from the crucible. Thus, the melting behavior may possibly change after long-term exposure to high temperature. Another crucible material (other than graphite) with mechanical and chemical inertness may prevent any compositional change.

In this work, alumina is used instead of graphite as another crucible material. An alumina crucible is chemically immune to reaction with graphite and many metals, even though it is somewhat more difficult to machine than common graphite. Previously alumina crucibles were used to construct pure metal high-temperature fixed points such as Ni and Pd [8,9]. In our first approach to using the new crucible, we selected the Fe–C system, which is known to have the lowest melting temperature of all the M–C systems. We also investigated its melting and freezing behaviors at various melting offset temperatures. Our goal in this work is to evaluate the possibility of using a ceramic material as a crucible material for a high-temperature fixed point.

2 Experimental Details

A double-wall alumina crucible and a thermometer well were prepared as shown in Fig. 1. Alumina crucibles were made using pure alumina powder having a purity of 99.8 %. The cell was 100 mm long and 33 mm in diameter. The wall thicknesses of the inner and outer crucible are 3.5 mm and 4 mm, respectively. The thermometer well had an external diameter of 14 mm and an internal diameter of 6 mm. The volume of the cell was about 8.25 cm³. An alumina protection tube with an outer diameter of 8 mm and an inner diameter of 6 mm was attached using alumina paste to the top of the thermometer well. About 54.5 g of Fe–C mixture was used as a filler in a pure Ar atmosphere. Pure Fe powder (99.998 %) and C powder (99.9999 %) were purchased from Alfa Aesar. When the powder was completely melted, the crucible and thermometer well were fitted together and then slowly cooled to room temperature. This cell assembly was inserted into one end closed alumina tube with an inner diameter of 35 mm. Alumina disc plates with a thickness of 0.5 mm were used as radiation shunts; they were supported by 30 mm long alumina tubes. After evacuating the tube to 1.3 Pa, pure argon (99.999 %) with a volume fraction of 0.5 % hydrogen was admitted to the cell. The internal pressure was kept slightly higher than an ambient level [2,4].

A type S thermocouple, which was calibrated at Ag, Cu, Fe–C, and Co–C fixed points, was used to monitor the melting and freezing behavior. The melting emf at the

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1 Protocol for the APMP regional comparison of Co–C eutectic melting point using Pt/Pd thermocouples.