Density Determination of Liquid Niobium by Means of Ohmic Pulse-Heating for Critical Point Estimation

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Abstract : Experimental determination of critical point data like critical temperature, critical pressure, critical volume and critical compressibility of high-melting metals such as niobium is very rare due to the outstanding experimental difficulties in reaching the necessary extreme temperature and pressure regimes. Experimental techniques to achieve such extreme conditions could be diamond anvil devices, two stage gas guns or metal samples hit by explosively accelerated flyers. Electrical pulse-heating under increased pressures would be another choice. This technique heats thin wire samples of 0.5 mm diameter and 40 mm length from room temperature to melting and then further to the end of the stable phase, the spinodal line, within several microseconds. When crossing the spinodal line, the sample explodes and reaches the gaseous phase. In our laboratory, pulse-heating experiments can be performed under variation of the ambient pressure from 1 to 5000 bar and allow a direct determination of critical point data for low-melting, but not for high-melting metals. However, the critical point also can be estimated by extrapolating the liquid phase density according to theoretical models. A reasonable prerequisite for the extrapolation is the existence of data that cover as much as possible of the liquid phase and at the same time exhibit small uncertainties. Ohmic pulse-heating was therefore applied to determine thermal volume expansion, and from that density of niobium over the entire liquid phase. As a first step, experiments under ambient pressure were performed. The second step will be to perform experiments under high-pressure conditions. During the heating process, shadow images of the expanding sample wire were captured at a frame rate of 4 × 105 fps to monitor the radial expansion as a function of time. Simultaneously, the sample radiance was measured with a pyrometer operating at a mean effective wavelength of 652 nm. To increase the accuracy of temperature deduction, spectral emittance in the liquid phase is also taken into account. Due to the high heating rates of about 2 × 108 K/s, longitudinal expansion of the wire is inhibited which implies an increased radial expansion. As a consequence, measuring the temperature dependent radial expansion is sufficient to deduce density as a function of temperature. This is accomplished by evaluating the full widths at half maximum of the cup-shaped intensity profiles that are calculated from each shadow image of the expanding wire. Relating these diameters to the diameter obtained before the pulse-heating start, the temperature dependent volume expansion is calculated. With the help of the known room-temperature density, volume expansion is then converted into density data. The so-obtained liquid density behavior is compared to existing literature data and provides another independent source of experimental data. In this work, the newly determined off-critical liquid phase density was in a second step utilized as input data for the estimation of niobium's critical point. The approach used, heuristically takes into account the crossover from mean field to Ising behavior, as well as the non-linearity of the phase diagram's diameter.

Keywords : critical point data, density, liquid metals, niobium, ohmic pulse-heating, volume expansion
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