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Recommended Specific Heat Capacity Functions of Group VA Elements

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The objective of this study was to investigate the possibility of representing specific heat capacities of the three metals comprising Group VA of the periodic table: vanadium, niobium and tantalum, with polynomials, from ambient temperature to close to their melting point temperatures. The analysis was based on available literature data including experimental studies of these metals at the Thermophysical Properties Laboratory of the "Vinča" Institute using millisecond resolution pulse calorimetry. This work has resulted in recommended functions obtained by analysis of existing experimental data. A critical analysis of methods used in obtaining these data, pointing to possible inherent sources of systematic errors that might influence their reliability, resulted in preferential weights of different data sets. Possible use of these three metals as candidates for specific heat capacity standard reference materials is also discussed.

KEY WORDS: calorimetry; high temperature; niobium; reference materials; specific heat capacity; tantalum; thermophysical properties; vanadium.

1. INTRODUCTION

Numerous technological applications require reliable specific heat capacity data. They are also needed for computing thermal transport properties, thermal conductivity or thermal diffusivity, as they are inter-linked via specific heat and density. Usually thermal conductivity is the one sought, although thermal diffusivity can be measured easier, particularly at high temperatures. For prediction of the specific heat capacity of alloys using the Kopp–Neumann law, an accurate knowledge of the specific heat capacity of constituent metals is necessary. For most of the above purposes, functions in the whole range are needed, generally from room temperature

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to melting points. Some applications require data in the liquid phase, or below room temperature.

There are various experimental techniques for measuring the specific heat capacity, none of them being equally reliable for different materials and in different temperature ranges. Yet, thermal property laboratories usually rely on one experimental technique, two at most, for the low- and high-temperature regions. It is, therefore, essential that materials with well established specific heat capacity values should be available, to enable testing of the performance of existing apparatus against reference data for particular types of materials in the temperature ranges of interest.

In the 1950s certain pure substances, including metals, have been selected and, through careful precision measurements, validated as calorimetry Standard Reference Materials, including recommended functions of their specific heats [1]. However, stocks of available thermophysical property SRMs have been generally depleted, and there has been no action toward their renewal, replacement or supplementing them with new materials (24th International Thermal Conductivity Conference 1997, Pittsburgh, Pa., conclusions of the Workshop on Reference Materials).

This paper represents an effort toward possible widening of the list of materials that might serve this purpose. Extensive experimental studies of calorimetric and electrical properties of refractory metals in the past decade at the Vinča Institute have provided new information on the calorimetric properties of these elements in a very wide temperature range, from room temperature to close to their respective melting points. The estimated maximum uncertainty of these results is 3%, which has been elaborated in Ref. 2 and summarized in Ref. 3. This maximum uncertainty applies to the upper and lower limits of the measurement range, i.e., within 150 K of the ends of the measurement range; between these extremes, the uncertainty is usually lower [4].

The results for experimental studies of three elements comprising Group VA at the Vinča laboratory have been presented in papers on vanadium [5], niobium [6], and tantalum [7]. A critical analysis of these results and available literature data provided recommended specific heat capacity functions, and also their potential use as calorimetry SRMs. In the following are presented the results of this analysis.

2. DISCUSSION

2.1. Theoretical Predictions

Experimental results for the specific heat capacity of refractory metals generally give higher values than predictions based on theoretical calculations. The difference between them increases with temperature, and is particularly evidenced as a strong nonlinear increase occurring at high temperatures. This phenomenon has been observed in our experimental studies of V [5], Nb [6], Ta [7], W [8], and Mo [9], as well as by other authors. In his review on equilibrium point defects in metals, Kraftmakher [10] discussed such behavior in specific heat capacity. Using tungsten as a typical example, he compared theoretical estimates [11] with the high temperature experimental data of Cezairliyan and McClure [12]. The difference between the highest positioned theoretical function referring to total isobaric specific heat, including vacancy contribution from positron-annihilation data [13], and experimental function of Cezairliyan and McClure [12] amounted to 10% at 2000 K; 19% at 2500 K; 29% at 3000 K, and reached as much as 54% at 3500 K. Different authors attribute this rapid upward trend of specific heat at high temperatures to vacancies [11], anharmonicity [14], anharmonicity and instability of crystal lattice [15], electronic effects [16], etc.; however, there is no general consensus or adequate quantitative interpretation which can account for such a difference. This applies not only to tungsten but to many refractory metals, including these in the Group VA. So, until new developments provide breakthroughs in theory with corresponding reliable quantitative predictions, recommended specific heat capacity functions must rely on reliable experimental results. Analysis/critical evaluation of the reliability of data of various authors, and how much weight they will be given for final averaging, depends heavily on the reliability of experimental methods applied in certain measurements and temperature regions.

2.2. Basic Groups of Experimental Methods

Below room temperature, adiabatic calorimetry is most frequently used as it provides very reliable results over the whole region. Above room temperature, there is a variety of methods which are convenient for metals: medium and high-temperature adiabatic and quasi-adiabatic calorimetry, pulse calorimetry, modulation calorimetry, and the method of mixtures, including levitation calorimetry. However, in this temperature range the use of methods that are not best suited for a particular purpose is likely to lead to errors, arising either from their inadequacy for studying particular materials in given temperature ranges, or from an inadequate interpretation of measured data. Reliability of adiabatic calorimetry decreases with increasing temperature, predominantly due to difficulties in maintaining and controlling adiabatic conditions when radiation heat exchange becomes more intensive. Nevertheless, variants of this method have been designed and used at temperatures exceeding 1300 K [17]. Methods in which the specimen is brought to equilibrium with the surroundings at a certain temperature level and then exposed to a small pulse of energy increasing its temperature by a few degrees [18] can be classified both as pulse and as quasi-adiabatic.

Pulse calorimetry avoids dangers of exposing the specimen to elevated temperatures for long periods and undesirable or uncontrollable heat exchange. The temperature is measured by high-speed pyrometry [19, 20], thermocouples [3], or the sample acting as a resistance thermometer [18]. Deficiencies of the first method [19, 20] are that the lowest operating temperature is limited to the lower limit of pyrometry, i.e., 1500 K [19] and 1000 K [20]. Also, in this method the thermal expansion of the specimen is limited; the specimen is held within a rigid sample holder that limits its longitudinal movement due to thermal expansion. In the method based on thermometry using thermocouples, the main difficulty arises from the large dc current passing through the specimen during the temperature measurement. This problem has been solved in Ref. 2 and presented in detail in Ref. 3. A simple way to check thermometry is via electrical resistivity measurements on the sample simultaneous with heating of the sample.

Modulation calorimetry, which consists of periodically modulating the power that heats the sample, creates temperature oscillations about a mean temperature. The amplitude of these oscillations depends on the heat capacity of the sample [21], so it is sufficient to measure the oscillations of the heating power and of the sample temperature. When the modulation frequency is sufficiently high, corrections for heat losses are negligible even at the highest temperatures. Modulation of the heating power can be caused by electrical current, thermal radiation, electron-bombardment heating, induction heating, use of separate heaters, Peltier heating, etc. The methods of detecting temperature oscillations include electrical resistance of the sample, or radiation from it, use of thermocouples, resistance thermometers, pyroelectric sensors etc. Modulation techniques allow measurements with high sensitivity over the widest temperature range, from fractions of a kelvin to the melting points of refractory metals. As a rule, the measurements are carried out in a regime where the amplitude of the temperature oscillations in the sample is inversely proportional to the heat capacity.

The method of mixtures gives enthalpy data as a primary outcome. For adequate fitting with a polynomial which must be differentiated to give the specific heat, the enthalpy data must be sufficiently numerous. The minimum number is taken to be 10, but for better accuracy of specific heat results, many more are necessary. This also implies that the temperature range over which enthalpy measurements are made has to be sufficiently large. Furthermore, the uncertainty of the enthalpy data depends on the uncertainty of the initial specimen temperature before the specimen is dropped into a calorimeter. It is usually established by measuring the temperature of the furnace in which the specimen is held assuming they are identical, what does not necessarily have to apply. The choice of polynomials for interpretation of enthalpy data introduces additional problems. In order to utilize fully the information contained in the enthalpy vs. temperature function many data points are needed, and for selecting the proper interpolating function, considerable experience and knowledge of the nature of specific heat is necessary [22]. A variant of the method of mixtures, levitation calorimetry, has an advantage that the sample temperature can be measured directly with a pyrometer, and the temperature intervals are usually sufficiently wide [23, 24].

3. RECOMMENDED SPECIFIC HEAT FUNCTIONS

3.1. Vanadium

Figure 1 presents the low-temperature data of Bieganski and Stalinski [25] and Anderson [26] which are not shown over their full range, and the

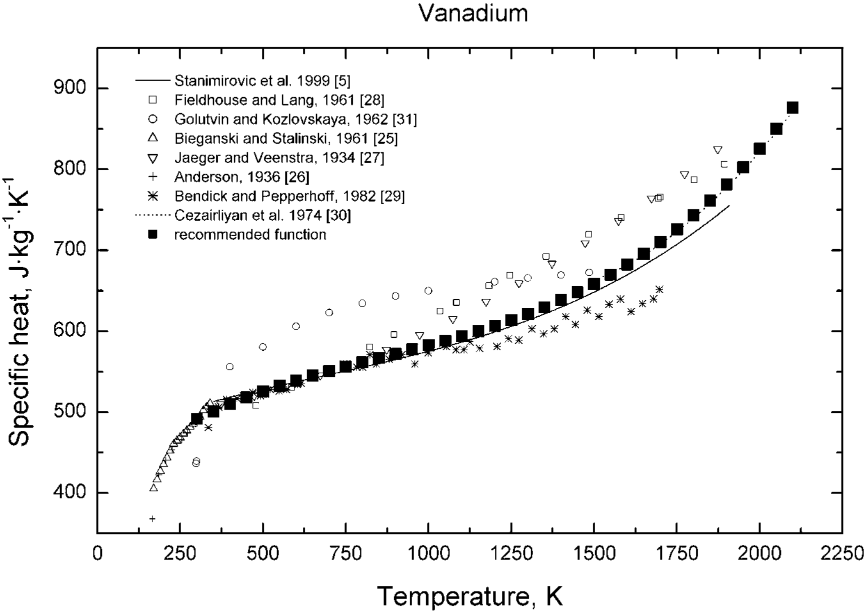

Fig. 1. Recommended specific heat capacity of vanadium.

specific heat data above room temperature of Jaeger and Veenstra [27], Fieldhouse and Lang [28], Bendick and Pepperhoff [29], Cezairliyan et al. [30], Golutvin and Kozlovskaya [31], and Stanimirović et al. [5]. The recommended function is presented at selected temperature intervals by full squares.

The two data sets of Bieganski and Stalinski [25] and Anderson [26] extend from low temperatures, the region where adiabatic calorimetry provides very reliable results, to above room temperature (340 K). They are in good agreement and can serve in defining the recommended function in the low-temperature range.

Between 300 and 1000 K there is reasonable agreement among the results of Stanimirović et al. [5] obtained with pulse calorimetry using thermocouple thermometry, the high temperature adiabatic calorimetry data of Bendick and Pepperhoff [29], and the results from the method of mixtures, computed from the measured enthalpy data of Jaeger and Veenstra [27] and Fieldhouse and Lang [28]. Above 1125 K the adiabatic calorimetry data [29] begin to fall below the other results, and the data sets computed from enthalpy results [27, 28] show different character, following either very mild parabolic [27] or linear [28] functions, both sets reaching their maximum values at 1880 K. Such shapes are frequently encountered in the behavior of specific heat data by drop calorimetry. The high-speed pyrometer pulse calorimetry results of Cezairliyan et al. [30] start at 1500 K, only 2.5% above the contact thermometry function [5], with similar behavior but with a somewhat steeper dependence with temperature. At 1900 K this difference reaches 3.6%. The maximum spread at 1690 K between the lowest [29] and the highest [27, 28] specific heat values is about 17%. The data of Golutvin and Kozlovskaya [31], obtained by metal-block drop calorimetry, have not been included as they differ from the rest both in character and in magnitude.

The recommended interpolation polynomial (1) follows the majority of the data to 1000 K, then continues slightly above the results from pulse calorimetry using thermocouple thermometry [5] to join the high-speed pyrometer pulse calorimetry results [30] from 1500 to 2100 K. Values calculated from Eq. (1) are in close agreement with the low-temperature data of Bieganski and Stalinski [25] and Anderson [26]. At 1690 K where adiabatic calorimetry data [29] end, it falls symmetrically between them and the highest drop calorimetry results [27, 28].

The recommended polynomial in the range 300 to 2100 K is given by the function,

$$Cp = 414.53974 + 0.32436T - 2.52541 \times 10^{-4} T^2 + 9.66295 \times 10^{-8} T^3 \quad (1)$$

3.2. Niobium

Figure 2 shows experimental results for niobium of Novikov et al. [32] obtained in the medium temperature range, Maglić et al. [6], and Kirillin et al. [23] which cover a wide temperature range, and the high temperature results of Righini et al. [20], Sheindlin et al. [24], Kraftmakher [33], Mozharov and Savvatimski [34], and Cezairliyan [35]. Most data in the latter group extend above the range of this study. In the range of 500 to 1800 K, the data of Maglić et al. [6], Kirillin et al. [23], Sheindlin et al. [24], and Novikov et al. [32] are in good agreement, of which the first used pulse calorimetry with thermocouple thermometry [6], the second [23] and the third [24] used "levitation" drop calorimetry, and the last used adiabatic calorimetry [32]. Above 1000 K they are joined by the results of Righini et al. [20] using pulse heating with scanning high-speed pyrometry. In the range above 1800 K, the data of Refs. 6, 23, and 24 are in good agreement while the data of Righini et al. [20] starting at 1000 K, fall about 1.5% below this common set. In parallel with these data are those of Cezairliyan [35], which are 1.6% below those of Ref. 20 at 1500 K, and 3.1% lower at 2500 K. The lowest two data sets are those of

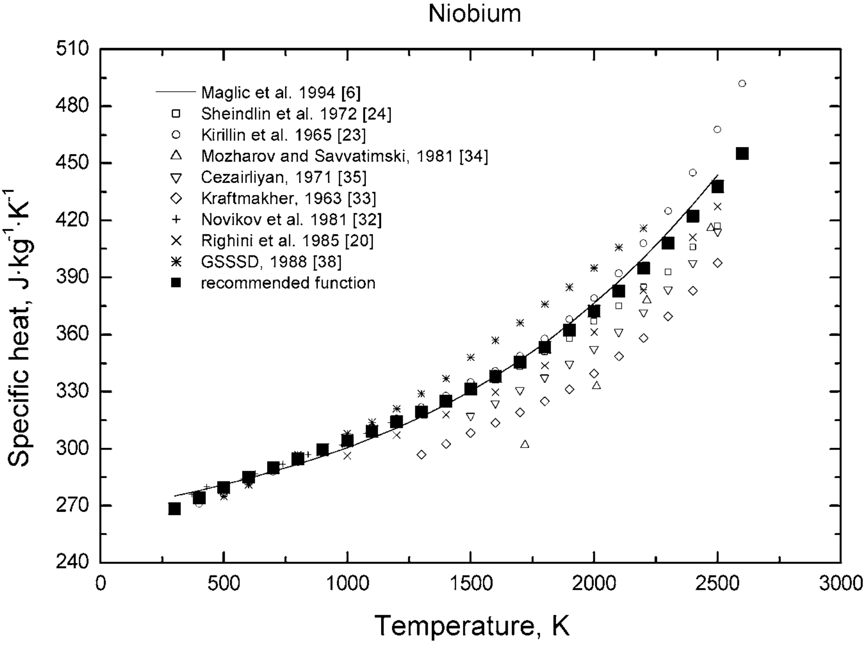

Fig. 2. Recommended specific heat capacity of niobium.

Kraftmakher [33] obtained by an equivalent-impedance modulation technique, and of Mozharov and Savvatimskii [34] obtained by an exploding wire method. According to information from Kraftmakher [36], the relatively low values of his specific heat function result from using literature data on the temperature derivative of the resistivity, $d\rho/dT$ [37], which are used in computation of the specific heats from direct measurements of $C/(d\rho/dT)$ using the equivalent-impedance technique. When more recent $C/(d\rho/dT)$ data of Righini et al. [20] are used, his specific heat values shift 4 to 6% higher, resulting in agreement within 1% with the Cezairliyan data [35]. Two points which stand out in the set of data at the lowest temperature of the exploding wire method measurements [34], are evidently not reliable, being at the at the low end extreme of the measurement range that reaches to 5000 K.

Similarly to vanadium, niobium also displays an upswing of its temperature function, although not so pronounced as in the case of the vanadium. The very recent results of Boboridis [39] carried out at NIST, Gaithersburg, Maryland, and published after this paper was completed further support the upswing behavior of the recommended function for niobium. His results at 1500 K start from the same level as these of Cezairliyan [35], but show a steeper increase with temperature.

In Fig. 2 is presented an interesting function which represents specific heat capacity data included in Soviet Tables of Standard Reference Data on Physical Properties of Niobium [38]. This report gives a specific heat function derived by computing it from standard reference values of thermal conductivity and thermal diffusivity, and density data. This function is more linear than the rest, lying not far above the mainstream.

In reaching the recommended specific heat capacity function of niobium, the two lowest temperature data points of Mozharov and Savvatimskii [34] were not used, and the data of Kraftmakher [33] were corrected according to the author's suggestion, i.e., by replacing the $C_p/(d\rho/dT)$ data of Reimann and Grant [37] with the corresponding data of Righini et al. [20]. After this, the spread of input data was about 5% at 1500 K, and reached about 12% at 2500 K. The GSSSD [38] data were not included.

The recommended specific heat capacity function easily joins with the literature low-temperature data of Clusius et al. [40] ranging from 10 to 273 K, which are in reasonable agreement at the low end with the data of Chou et al. [41] from 1.3 to 29 K.

The specific heat capacity of niobium in the range 300 K to 2500 K is represented by the function,

$$Cp = 246.74642 + 0.08211T - 3.94862 \times 10^{-5}T^2 + 1.48903 \times 10^{-8}T^3 \quad (2)$$

3.3. Tantalum

Figure 3 presents available experimental results of the specific heat capacity of tantalum above room temperature. The widest temperature region was covered by Taylor and Finch [18] and Lehman [42] for which the data cover a range from 100 to 3200 K. The results of Kulish and Philippov [43] range between 300 and 500 K, and these of Milošević et al. [7] between room temperature and 2300 K. In the high temperature range the Taylor and Finch [18], Lehman [42], and Milošević et al. [7] data connect with the data of Cezairliyan et al. [44], Rasor and McClelland [45], Lowenthal [46], and Hoch and Johnston [47]. The nine data sets were obtained using different measurement techniques. Taylor and Finch [18] and Rasor and McClelland [45] used a variant of the sample resistance method, Milošević et al. [7] used a variant of thermocouple thermometry, and Cezairliyan et al. [44] used a high-speed pyrometer variant of the pulse method; Lowenthal [46] and Kulish and Philippov [43], applied a method of temperature oscillations; and Sterrett [49] and Hoch and Johnston [47] used the method of mixtures, the latter of them using high-temperature vacuum drop calorimetry.

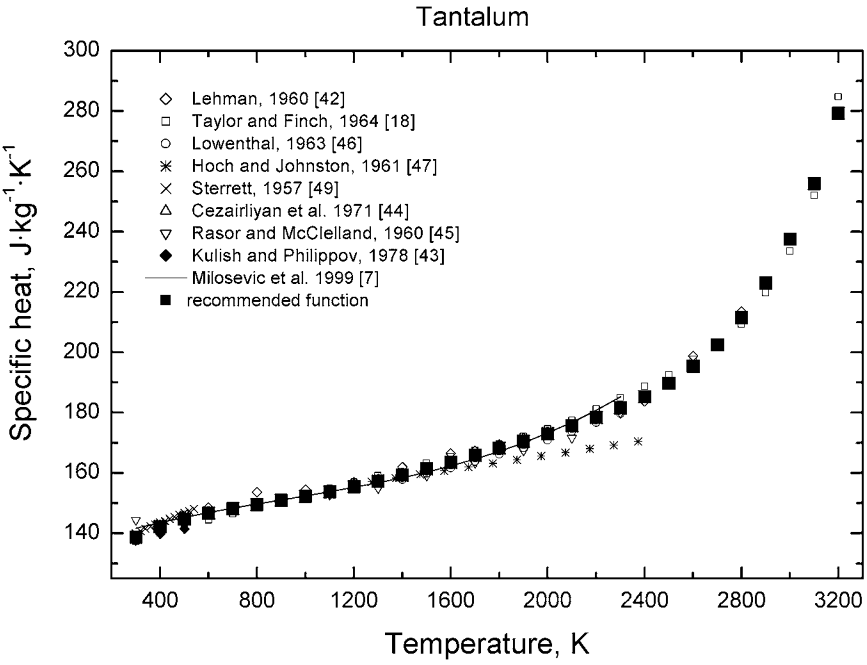

Fig. 3. Recommended specific heat capacity of tantalum.

Agreement among results is generally good. With the exception of the Hoch and Johnston [47] data, all experimental results fall within a narrow belt whose width is about 5% at room temperature, which is reduced to about 3.5% at 1500 K. The linear Hoch and Johnston [47] data show less dependence on temperature than the other methods, this is likely due to selecting a second order polynomial for fitting their high-temperature enthalpy data.

In defining the proposed recommended specific heat function of tantalum, all data could be used except these of Hoch and Johnston [47] for the reason discussed above. Care was taken to ensure a smooth transition to the low-temperature data near room temperature. In the region 1.3 to 24 K, the specific heat capacity of tantalum is represented by the data of White et al. [48], between 15 and 539 K by those of Sterrett [49]. All of them, including the low temperature portions of the Taylor and Finch [18] and Lehman [42] results which start from 100 K are in reasonable agreement and could provide a base for defining a recommended function in the low temperature region. Above room temperature, the wide range results of Taylor and Finch [18] and Lehman [42] are supported with results from Refs. 7, 43, 44, 45, and 46. The data of Sterrett [49] above room temperature, have also been included in the fit.

The specific heat capacity of tantalum in the range 300 to 3200 K can be represented by the function,

$$\begin{aligned} Cp = {} & 117.989 + 0.10524T - 1.5259 \times 10^{-4} T^2 + 1.17931 \times 10^{-7} T^3 \\ & - 4.20425 \times 10^{-11} T^4 + 5.75517 \times 10^{-15} T^5 \end{aligned} \tag{3}$$

According to the Dulong–Petit law, the specific heat of tantalum should enter a region of saturation above the Debye temperature which, for this metal, lies at about 230 K, changing magnitude by only 30% over the next 2000 K. At elevated temperatures, however, experimental results of virtually all researchers exceeded such a prediction. A similar tendency has also been observed in the final functions of the specific heat of vanadium and niobium, as well of molybdenum [9] or tungsten, discussed in Section 2.1. These higher than predicted values in tantalum have been attributed to electronic contributions to the specific heat [46], electronic contributions and thermal formation of lattice imperfections at high temperatures [45], equilibrium point defects [10], or other possible causes [18]. A recent review on equilibrium point defects and thermophysical properties of metals [10] discusses this phenomenon at length.

3. CONCLUSIONS

All three metals proved convenient to work with, with regard to low oxidation under not too high vacuum conditions and maintaining their geometry on exposure to high temperatures. The general consensus within given groups of data for particular metals allowed proposing recommended functions of specific heat capacity vs. temperature over large intervals. Agreement between our results and literature data are satisfactory.

A choice of one or more of them as a specific heat capacity SRM depends upon a number of factors such as the width of the temperature range, cost, resistance to oxidation, etc. However, all of them seem to be more convenient than tungsten with respect to workability of the material, and tendency to grain growth, at least within our measurements.

The character of the specific heat capacity of the three metals of the Group VA at high temperatures approaching the melting range did not follow theoretical predictions, demonstrating a distinct upswing, similar to other refractory metals. Theoretical studies should offer acceptable explanations with more consensus among them.

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