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High Temperature Heat Capacity of Alloy D9 Using Drop Calorimetry Based Enthalpy Increment Measurements

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Alloy D9 is a void-swelling resistant nuclear grade austenitic stainless steel (SS) based on AISI type 316-SS in which titanium constitutes an added predetermined alloying composition. In the present study, the high-temperature enthalpy values of alloy D9 with three different titanium-to-carbon mass percent ratios, namely Ti/C = 4, 6, and 8, have been measured using inverse drop calorimetry in the temperature range from 295 to 1323 K. It is found that within the level of experimental uncertainty, the enthalpy values are independent of the Ti–C mass ratio. The temperature dependence of the isobaric specific heat C_P is obtained by a linear regression of the measured enthalpy data. The measured C_P data for alloy D9 may be represented by the following best-fit expression:

$$C_P(\mathrm{J\cdot kg^{-1}\cdot K^{-1}}) = 431 + 17.7\times10^{-2}T + 8.72\times10^{-5}/T^2.$$

It is found that the measured enthalpy and specific heat values exhibit good agreement with reported data on 316 and other related austenitic stainless steels.

KEY WORDS: alloy D9; austenitic stainless steel; drop calorimetry; enthalpy; heat capacity.

1. INTRODUCTION

In view of their good high temperature mechanical properties, oxidation resistance, weldability, and excellent compatibility with liquid sodium, austenitic stainless steels are the preferred candidates for making most of the

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core components of liquid-metal-cooled fast reactors [1]. But one major concern with the wide spread use of austenitic steels in a highly radioactive environment is their poor void-swelling resistance against fast neutron irradiation [2]. Keeping in line with the current understanding of void-swelling phenomena [3], certain alloy design strategies have been adopted in the past to improve the neutron void-swelling resistance of standard 18–8 type austenitic stainless steels [4–9]. Some of these include (i) a slight enhancement of the nickel-to-chromium content ratio, along with a controlled addition of minor alloying constituents such as silicon that serve to bind the vacancy-impurity complexes [4], (ii) promotion of radiation-induced precipitation that alters the migration characteristics of point defects [5], (iii) addition of titanium, again in small quantities (less than one mass%) to facilitate the formation of fine TiC precipitates whose interface with the matrix serves as sinks for the radiation-induced defects [6], (iv) a certain amount of prior-deformation or cold working of the steel, which by virtue of providing a dislocation strewn matrix microstructure, contributes to the annihilation of irradiation-induced point defects [7], and (v) explicit addition of a small quantity of phosphorous to form phosphide plates whose interface with the *fcc*-austenitic matrix is believed to be conducive for nucleating helium bubbles that in turn absorb the radiation-induced vacancies [8, 9]. Thus, by playing around with one or more of such alloy design considerations, a few nuclear grade austenitic stainless steels have been designed from existing standard varieties.

The second phase of the Indian Nuclear Power Programme involves the construction of a 500 MW(*e*) Prototype Fast Breeder Reactor, and for this reactor a specific variety of austenitic stainless steel alloy D9 has been chosen as the prospective core structural material [10]. The nominal composition of this steel is presented in Table I. It may be said that the composition of D9 is derived from that of conventional 316-SS by slightly modifying the Ni, Cr, Si, Ti, and C contents. This point is also brought out in Table I, wherein, for the purpose of comparison, we have also listed the compositions of other standard austenitic stainless steels. In alloy D9, titanium constitutes an explicit alloying addition. Furthermore, a prior cold work of about 20% constitutes a key material specification [10]. The relative amounts of titanium to carbon is adjusted in such a way that three different compositional variants of D9 with Ti/C $\approx$ 4, 6, and 8 have been made, with an effort to study the effect of the Ti/C ratio on mechanical and other physical properties.

Although an extensive characterization of the in-pile behavior of this material is rather necessary for enabling a knowledge base design of reactor core components, it is essential to stress the fact that comprehensive out-of-pile characterization studies constitute a vital prerequisite

Table I. Nominal chemical composition[a] (in mass percent) of alloy D9 from present study, together with compositional details of other related austenitic stainless steels. (It must also be stated that in general there is considerable scatter in the stated compositions of the same steel by different investigators and what is quoted here are those compositions for which experimental thermophysical property data are available.)

Element	304[b]	304L[d]	316[b]	316LN[e]	321[c]	D9[a]	D9[f]
Ni	9.7	9.3	11.7	12.5	9.0–12.0	14.9±0.5	15.5
Cr	18.4	18.5	16.8	18.0	17.0–19.0	14.7±0.5	13.5
Mn	1.4	1.16	1.9	1.6–2.0	<2.00	1.3±0.05	2.0
Mo		0.15	2.1	2.7		2.2±0.05	2.0
Cu		0.1	0.2	1.0		<0.050	
Ti					$\geqslant 5\times\%\mathrm{C}$	0.18±0.006	0.25
Nb						<0.07	
V						0.045±0.005	
Co		0.18		0.25		0.03±0.005	
Al						<0.034	
Sn						<0.004	
W						<0.005	
Si	0.6	0.7	0.4	0.05	$\leqslant 1.00$	0.65±0.05	0.75
C	0.02	0.022	0.05	0.03	$\leqslant 0.08$	0.05±0.005	0.04
N		0.010		0.08		<0.04	
P	0.02	0.010	0.03	0.035	$\leqslant 0.045$	0.008±0.001	
S	0.01	0.011	0.02	0.025		0.005±0.003	
B				0.002			
As						<0.006	
Fe	balance	balance	balance	balance	balance	balance	
Density at ~ 300 K (kg $\cdot$m^{-3})	~7860	~8000	~7970	~7966		~ 8200[g]	

[a] Determined in this study using direct reading optical emission spectrometry.

[b] From Ref. 31.

[c] From Ref. 20.

[d] From Ref. 22, 23.

[e] From IGCAR internal report (PFBR/01000/DN/1000).

[f] From Ref. 29.

[g] Density determined in the present study using immersion technique.

in this direction. Accordingly, a broad-based thermal, mechanical, and physical property characterization of this steel has been initiated and a part of which, namely the mechanical property evaluation, has already been completed [10–14]. In addition, the high-temperature lattice thermal-expansion characteristics of a specific variety of D9 with Ti/C = 6 have been investigated by us rather recently [15]. The present study is concerned with the out-of-pile measurement of high-temperature enthalpy values and thereby the specific heat of indigenously developed alloy D9. In particular, the enthalpy values of three compositional variants, with Ti/C ≈ 4, 6, and 8 have been measured using a drop-calorimetric technique in the temperature range from 300 to 1373 K. The results of this study are reported in this paper.

2. EXPERIMENTAL DETAILS

2.1. Material

The D9 stainless steel used in the present study was procured from MIDHANI, India. The original material, melted by a vacuum induction technique, was obtained in the form of forged rods of varying diameter. These are subsequently solution annealed at 1323 K for about an hour in an argon inert gas environment. The compositional analysis has been performed using wet-chemical and direct reading optical emission spectrometry with the aid of appropriate elemental standards. The nitrogen content has been determined separately by an inert gas fusion technique. For all three D9 varieties, varying the titanium-to-carbon content ratio, the nominal composition is the same with respect to the major elements. This nominal composition is presented in Table I. It must be noted that there is some scatter in the composition of steel samples coming from different heats. However, the composition listed here pertains to that particular lot from which the samples for drop calorimetry measurements have been drawn. The bulk density of the solution-annealed samples has been determined by means of a standard immersion technique using Armco iron as the density standard. The representative value of the density of D9 steel used in this study is found to be $7.997\,\mathrm{g{\cdot}cm^{-3}}$. There is a small spread (7.9–$8.2\,\mathrm{g{\cdot}cm^{-3}}$) in the nominal density values of the three Ti/C compositions. This density spread is however ignored here. The room temperature (295 K) x-ray diffraction profile revealed the presence of only *fcc* austenite, and its lattice parameter is found to be 0.3957 ± 0.0007 nm.

2.2. Drop Calorimetry Measurements

The enthalpy increment ($H_T - H_{295}$) with respect to the chosen reference temperature (295 K) has been measured by the Setaram—MHTC 96® calorimeter operating in the drop mode. The samples for drop calorimetry are obtained by cutting small pieces from solution-annealed D9 rods. The mass of the samples, which varied from 75 to 100 mg is accurately determined by a precision balance to ±0.01 mg. The working chamber of the drop calorimeter consists of a long ceramic (Al_2O_3) tube, which houses a small volume ($5.3\,cm^3$) alumina crucible at its bottom. This crucible is filled to two-thirds of its volume with pure dry alumina powder supplied by Seteram. This alumina-filled ceramic crucible forms the actual drop bed. The temperature of the bed is sensed accurately by an annular array of S-type thermocouples (Pt-PtRh10%) positioned circumferentially around the bed. This serves to provide an integrated and efficient means of sensing the changes in temperature of the bed. The drop tube sensor is heated by a surrounding graphite furnace.

During the course of performing a drop experiment, previously weighed samples of alloy D9 together with α-alumina standards are loaded alternately into distinct slots provided in the specimen-feeding chamber, which is located at the top of the drop tube sensor. To begin with, the working chamber is evacuated using a rotary pump and is subsequently flushed with pure argon. In fact, the whole experiment consisting of specimen dropping followed by recording of the change in the temperature of the bed is performed under the continuous flow of argon. The drop bed is gradually heated to the preset temperature at the rate of $10\,K\cdot min^{-1}$. After the bed has attained a reasonable degree of thermal equilibrium, which is attested by the constancy of its temperature to within $\pm 0.1\,K$, the sample is dropped from the feeding chamber into the bed under isothermal conditions. The heat absorbed by the specimen upon its drop from the ambient temperature into the hot alumina bed is accurately quantified by monitoring the quantum of heat flux $\phi(\mu V)$ as a function of time t (s) by means of the proprietary data acquisition software provided by Seteram. In the initial runs performed on $Ti/C=4$ samples, we performed four individual drop experiments for each temperature step. This is necessary to gauge the typical spread in the measured enthalpy values, which is about 8% for higher temperatures ($T \geqslant 1200K$). It must be noted that at high temperatures, some mild oxidation of the sample did occur and this might have contributed to the scatter observed in the high-temperature enthalpy data. During the course of these experiments, each of the sample droppings is alternated by an α-alumina standard under identical experimental conditions. A time interval of about 30 min is provided between the drops as this is found to be adequate for the restoration of thermal equilibrium of the bed.

Assuming no heat loss due to radiation, the time-averaged quantum of heat flux is given by the expression [16],

$$Q_S(T) = \int \phi_S dt = (H_T - H_{295})_S (m_S/M_S) C. \tag{1}$$

In the above expression, $(H_T - H_{295})_S$ represents the enthalpy increment of the sample with respect to the reference temperature 295 K, (m_S/M_S) is the ratio of the actual mass of sample to its molar mass, and C is a calorimeter sensitivity parameter. $Q_S(T) = \int \phi_S \mathrm{d}t$, is the time averaged heat flux that is measured as the total area under the curve of a baseline corrected temperature *versus* time profile. This measurement is facilitated automatically by the proprietary data acquisition and processing software supplied by Seteram. The calorimeter constant C may be determined as a function of temperature by measuring $\int \phi_R \mathrm{d}t$, for the α-alumina standard under identical experimental conditions. It must be noted that for α-alumina, critically assessed enthalpy data are available in the literature [17]. Thus, for the α-alumina drop experiment, we may again express the heat flux in the following manner:

$$Q_R(T) = \int \phi_R \mathrm{d}t = (H_T - H_{295})_R (m_R/M_R) C. \tag{2}$$

From Eq. (2) the quantity C can be determined by knowing $Q_R(T)$ and $(H_T - H_{295})_R$. Now, in view of Eqs. (1)and (2), it emerges that the enthalpy increment $(H_T - H_{295})_S$ of the sample at any temperature T is given by

$$(H_T - H_{295})_S = (H_T - H_{295})_R \times \{Q_S/Q_R\} \times \{(M_S/m_S)/(M_R/m_R)\}. \tag{3}$$

In the above expression, $\{Q_S/Q_R\}$ is the experimentally measured quantity. $(\mathrm{H}_T - H_{295})_R$ is taken from the literature. Thus, $(H_T - H_{295})_S$ can be estimated for a known mass ratio of the sample and enthalpy standard. It must be stressed that the calibration of the calorimeter sensitivity parameter C is done simultaneously and at the same experimental conditions as that of the specimen enthalpy measurement.

3. RESULTS

3.1. Enthalpy and Specific Heat of D9

In Fig. 1, the enthalpy increment data are plotted with respect to temperature for the three Ti/C compositions. For the sake of comparison, we have also included from the literature, data for 316 and 347 varieties [18–20]. From Fig. 1, the following points emerge. There is reasonable agreement among the enthalpy increment values of three D9 compositions. It may be said that in the temperature range of our measurement, the measured enthalpy of D9 is independent of the Ti/C ratio. Moreover, the D9 enthalpy data obtained in the present study show an overall agreement with the values for 316 and 347 stainless steels [18–20].

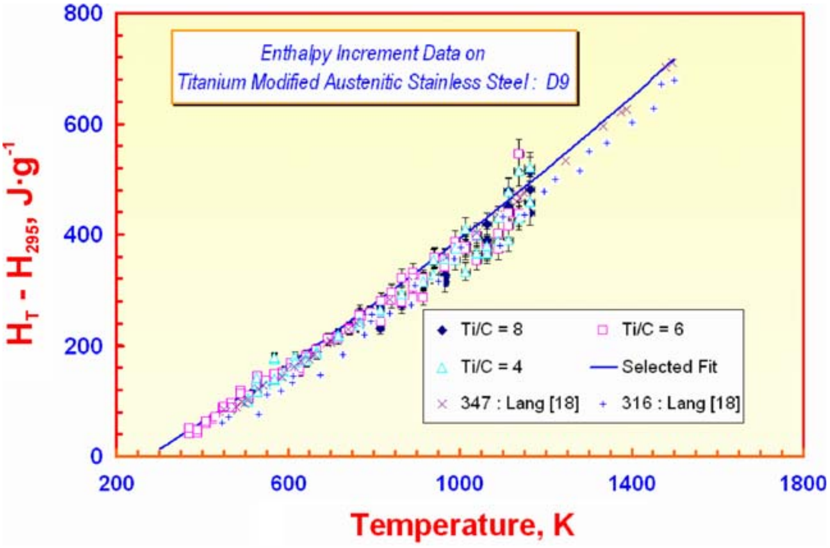

Fig. 1. Measured enthalpy values for alloy D9 are plotted together with the corresponding data on 316 and 347 stainless steels, taken from the literature [18–20].

The consolidated enthalpy data obtained in the present study on all three D9 compositions may be represented by the following expression

$$H_T - H_{295} = -1.24 \times 10^2 + 4.31 \times 10^{-1} T + 8.85 \times 10^{-5} T^2 - 8.72 \times 10^{-8}/T. \quad (4)$$

The R^2 value of the above fit is 0.98. In the above expression $H_T - H_{295}$ is given in J·g^{-1} and T in K. The corresponding isobaric specific heat C_P is given by the expression,

$$C_P = 431 + 17.7 \times 10^{-2} T + 8.72 \times 10^{-5}/T^2. \quad (5)$$

In Eq. (5), C_P is given in J·kg^{-1}·K^{-1}. From Eq. (5), a C_P value of about 484 J·kg^{-1}·K^{-1} is obtained for $T = 298$K. This value is fairly close to the value of 472 J·kg^{-1}·K^{-1} listed by Harding [21] for 316 stainless steel. It must be mentioned that a spread of about 8% is noticed between diverse estimates of C_P at 298 K for 316, 321, 347, and 304L type austenitic stainless steels [18–24]. The present estimate of 484 J·kg^{-1}·K^{-1} for D9 has been found to be well within this band (Fig. 2). In addition, it is also evident that the measured high-temperature C_P data of D9 merges fairly smoothly with the recommended low-temperature specific-heat data of 316 stainless

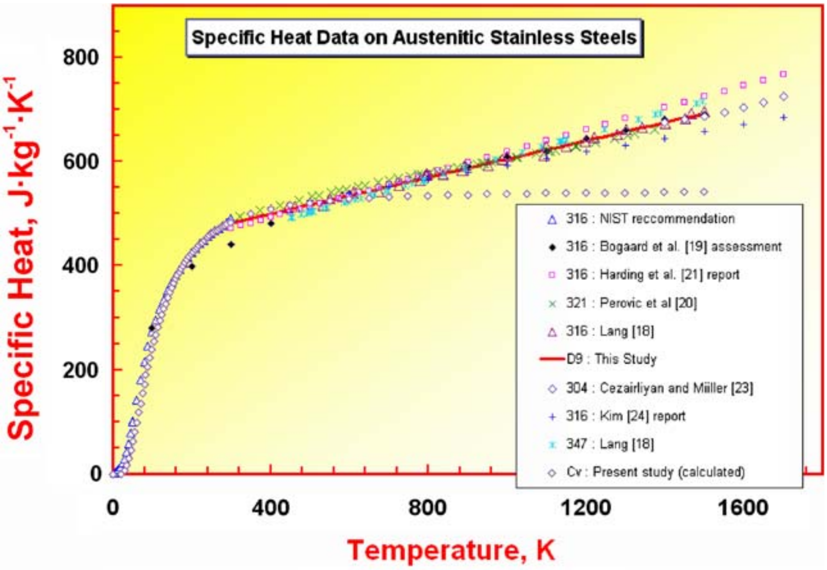

Fig. 2. Specific heat C_P of alloy D9 estimated from the measured enthalpy values is plotted as a function of temperature. C_P data for other austenitic stainless steels are taken from the literature [18–20]. C_V is calculated from C_P using experimental thermal expansion data [15] and an estimated temperature-independent value for the Grüneisen parameter.

steel (Fig. 2) [25,26]. In Table II, the smoothed values of enthalpy and specific heat as given by Eqs. (4) and (5), respectively, are listed.

3.2. Estimation of C_V

From thermodynamics, the following relationship between C_P and C_V, the isochoric specific heat is obtained [27]:

$$C_P = C_V(1 + \gamma_G \alpha_V T). \tag{6}$$

In the above expression, γ_G is the thermal Grüneisen parameter and α_V is the coefficient of volume thermal expansion. As noted before, the lattice thermal expansivity of D9 with a Ti/C of 6 has already been determined using high-temperature x-ray diffraction [15]. At the moment it is not clear as to whether the thermal expansivity of alloy D9 is independent of the relative amounts of titanium to carbon. In the absence of reliable experimental data to this effect, we take the measured thermal expansion values of Ti/C = 6 to hold good for all three D9 grades. As far as γ_G is concerned, again there are no experimental data available, primarily due to

Table II. Enthalpy increment ($H_T - H_{295}$) and specific heat, C_P values of alloy D9 obtained in present study. (Experimental data are obtained only up to 1323 K, but are extrapolated to 1500 K using the best-fit expression.)

T	$H_T - H_{295}$	C_P
(K)	(10^3 J·kg^{-1})	(J·kg^{-1}·K^{-1})
298	12.30	483.75
300	13.26	484.10
350	37.69	492.95
400	62.56	501.80
450	87.87	510.65
500	113.62	519.50
550	139.82	528.35
600	166.46	537.20
650	193.54	546.05
700	221.06	554.90
750	249.03	563.75
800	277.44	572.60
850	306.29	581.45
900	335.58	590.30
950	365.32	599.15
1000	395.50	608.00
1050	426.12	616.85
1100	457.18	625.70
1150	488.69	634.55
1200	520.64	643.40
1250	553.03	652.25
1300	585.86	661.10
1350	619.14	669.95
1400	652.86	678.80
1450	687.02	687.65
1500	721.62	696.50

the absence of elastic property data. In the absence of experimental information on alloy D9, we use a constant (temperature-independent) value of 1.6 for γ_G that is obtained from shock-wave studies on 316 stainless steels [28]. Using these data, C_V has been estimated from the measured C_P, and these values are plotted in Fig. 2. It is clearly seen that at high temperatures, C_V tends to attain a limiting value.

It is also possible to get an estimate of the lattice contribution to C_V by invoking the standard Debye model. For this purpose, we assumed a θ_D value of 450 K, which is based on our recent lattice thermal expansion data of D9 with Ti/C = 6 [15]. An average molar mass of 56.41 is used in affecting the conversion of the calculated C_V from a per mole to a per kg basis [15]. Discounting the relatively small electronic contribution to the specific heat, the Debye model C_V agreed to within about 2% of the values obtained from experimental C_P and α_V via Eq. (6). In order to avoid unnecessary clutter, the calculated Debye C_V is not presented in Fig. 2.

4. DISCUSSION

At the outset, it is useful to clarify a few general points concerned with this study. First, one must note that although austenitic stainless steels are one of the most widely used engineering materials, there are not many recent references to their high-temperature thermophysical properties. This situation is especially true for nuclear grade steels. For popular varieties like 304, 304L, 316, 321, etc., reliable out-of-pile experimental thermal property data do exist, as a result of some classical studies [18–20,22,23]. In addition, Harding et al. [21] and Kim [24] have reviewed the general thermophysical property situation in austenitic stainless steels. Blomquist and Leibowitz [29] have measured the high-temperature bulk thermal expansion of D9 (American version, see Table I) using dilatometry. It is amidst this relative paucity of recent data, the present authors have initiated a course of study on thermal property characterization of the Indian version of alloy – D9. The second point is that the present study is oriented towards generating accurate out-of-pile data, although it is expected that considerable long-term exposure to fast neutron flux during the course of in-pile service modifies the overall thermophysical property characterization, by giving rise to considerable defect-induced contributions. As mentioned in the introduction, since an accurate out-of-pile characterization serves as a benchmark for assessing irradiation effects on properties, the scope of the present study is restricted to generating baseline enthalpy values of unirradiated D9 alloy. The thermal property characterization of irradiated alloy D9 is deferred to a future study.

As far as the present study goes, the measured enthalpy of alloy D9 is of the same order of magnitude as that of typical 316 type austenitic steel. This is despite the fact that alloy D9, even in the solution annealed state, contains a very fine dispersion of TiC particles in the austenitic matrix [30]. However, their presence is not revealed in the corresponding X-ray diffraction profile. Therefore, it is clear that the phase fraction of TiC must be rather small to significantly register their effect in the measured enthalpy values. Probably, this is the reason as to why the measured C_P is very close to that of typical 316 stainless steels. It is interesting to note that the present C_P estimate for alloy D9 is similar to that of 321, another titanium modified stainless steel [20]. Another important finding of this study is that we could not detect any meaningful dependence of enthalpy on the ratio of titanium to carbon content. In all likelihood, we believe that the phase fraction of TiC is not appreciably different in the range of Ti/C investigated here. It would be rather interesting to see whether such behavior is echoed by the thermal expansion characteristics of alloy D9 as well. Further studies in this direction are underway.

5. CONCLUSIONS

The high-temperature heat capacity of alloy D9 with Ti/C = 4, 6, and 8 has been measured using drop calorimetry. The measured C_P results agree well with the values expected for 316-based austenitic stainless steels. No dependence of C_P with the Ti/C ratio is evident from the present study.

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