Measurement of Thermal Diffusivity of Steels at Elevated Temperature by a Laser Flash Method

Tsuyoshi NISHI, Hiroyuki SHIBATA,¹⁾ Koichi TSUTSUMI,²⁾ Hiromichi OHTA³⁾ and Yoshio WASEDA¹⁾

Graduate School, Department of Materials Processing, Tohoku University, Sendai 980-8577 Japan.

E-mail: nishi@iamp.tohoku.ac.jp. 1) Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Sendai 980-8577 Japan. 2) NKK Corporation, Materials and Processing Research Center, Fukuyama, Hiroshima 721-8510 Japan. 3) Department of Materials Science, Ibaraki University, Nakanarusawa, Hitachi 316-8511 Japan.

(Received on December 27, 2001; accepted in final form on February 2, 2002)

In order to obtain thermal diffusivity of steels at elevated temperature with sufficient reliability using a laser flash method, an accurate value of specimen thickness is essentially required. Accordingly, a linear thermal expansion coefficient of steels was systematically measured in the temperature range from room temperature to 1676 K. Since the decrease in specimen thickness was detected after the measurement of thermal diffusivity under vacuum at high temperature close to solidus, such a factor was quantitatively estimated from the thickness values of quenched specimens. Combining these two results, the thermal diffusivity values were successfully determined for ultra low carbon, low carbon, medium carbon, 1.2 % silicon, 9 % nickel and 13 % chromium steels in the temperature range from room temperature to 1676 K using the laser flash method.

KEY WORDS: thermal diffusivity; elevated temperature; thickness of specimen; steel; linear thermal expansion coefficient; laser flash method.

1. Introduction

Thermal diffusivity values of steels at elevated temperatures are important to control continuous casting and hot rolling processes. Computer simulation with a model is frequently used and such works are quite useful to improve the productivity and the energy saving by optimizing the processes. In these simulation studies, accurate values of thermal properties as a function of temperatures are strongly required to improve the reliability of the model. However, the thermal diffusivity values of steels with sufficient reliability are not available at high temperature, particularly at temperature above 1400 K,¹⁾ because of experimental difficulties. On the other hand, a laser flash method is well recognized as a powerful tool for measuring thermal diffusivity of a solid specimen at elevated temperature. In this laser flash measurement, an accurate value of the specimen thickness at desired temperature is essentially required to determine thermal diffusivity. However, the linear thermal expansion values of steels are not available at high temperature.²⁾ In addition, the experimental uncertainty of the thermal diffusivity value of steels is also affected by the change in specimen thickness at temperature near solidus under vacuum.

In order to overcome these difficulties, the linear thermal expansion values of steels are measured in the temperature range from room temperature to solidus. In addition, the variation of the specimen thickness with sufficient reliability is estimated from the thickness values of quenched speci-

mens. Then, the laser flash method is also applied to three carbon steels (ultra low carbon, low carbon, and medium carbon steels) and three special steels (1.2% silicon, 9% nickel and 13% chromium steels). This includes the variation of the specimen thickness during the course of measurement.

The main purpose of this work is to obtain the thermal diffusivity values of commercial steels with typical chemical composition at high temperatures close to solidus systematically and to provide the recommended thermal diffusivity values as a function of temperature.

2. Experimental Method

Chemical compositions and some relevant properties of six steel specimens are summarized in **Table 1**. The per-

Table 1. Chemical composition (unit: mass%), liquidus (TL: K) and solidus (TS: K) for ultra low carbon steel (ULCS), low carbon steel (LCS), medium carbon steel (MCS), 1.2% silicon steel (1.2% Si), 9% nickel steel (9% Ni) and 13% chromium steel (13% Cr).

composition	ULCS	LCS	MCS	1.2 % Si	9 % Ni	13 % Cr
C	0.00017	0.028	0.135	0.0012	0.05	0.2
Si	0.01	0.01	0.32	1.23	0.25	0.27
Mn	0.17	0.2	1.35	0.29	0.58	0.48
P	0.019	0.008	0.014	0.041	0.002	0.009
S	0.008	0.006	0.007	–	0.001	0.001
sol. Al	0.038	0.029	0.026	0.3	0.042	0.026
temperature	ULCS	LCS	MCS	1.2 % Si	9 % Ni	13 % Cr
TL ⁽¹⁾	1807	1806	1799	1790	1773	1763
TS ⁽¹⁾	1793	1781	1758	1759	1723	1703

Fig. 1. Schematic diagram of a laser flash technique.

centage linear thermal expansion of the six steel specimens were measured in the temperature range from room temperature to 1 676 K under an argon gas flow condition by using a dilatometer. The size of steel specimens for measurement of linear thermal expansion is 25 mm in length and 5 mm in diameter.

Thermal diffusivity was measured under vacuum of less than 3×10^{-3} Pa by a laser flash method. As shown in Fig. 1, the upper surface of a specimen with a disk shape, 1 mm in thickness and 10 mm in diameter, was instantaneously irradiated by Nd glass laser (10 J, 1 060 nm emission wavelength). Then, the temperature response at the bottom surface of the specimen was measured by using an InSb infrared detector (1.2–5.5 μm effective wavelength). The specimens were heated up to 1 676 K with a tungsten mesh heater. The specimen surface was coated with a graphite powder spray to improve the signal to noise ratio of the response curve at room temperature. On the other hand, the graphite powder spray was not utilized at elevated temperatures to keep the specimens free from contamination with carbon. In addition, the signal to noise ratio was improved in this work by accumulating ten temperature response curves.

The half-time method³⁾ at elevated temperature in the temperature range of above 1 000 K is known to be, more or less, affected by radiative heat loss from the specimen surface. Then, the curve fitting method was employed in this work to determine the thermal diffusivity value of the steel specimens. The temperature rise of the rear surface, T_r , is described by Eq. (1).

$$T_r = T_M \sum_{m=0}^{\infty} A_m \exp \left[- \left(\frac{X_m}{\pi} \right)^2 \frac{t}{t_0} \right] \quad \text{.....(1)}$$

$$t_0 = l^2 / (\pi^2 \alpha) \quad \text{.....(2)}$$

$$A_m = 2(-1)^m X_m^2 (X_m^2 + 2Y + Y^2)^{-1} \quad \text{.....(3)}$$

$$X_0 = (2Y)^{1/2} (1 - Y/12 + 7Y^2/288) \quad \text{.....(4)}$$

$$X_m = m\pi + 2Y/m\pi - 4Y^2/(m\pi)^2 - 2Y^3/3(m\pi)^3 + 16Y^4/(m\pi)^4 \quad \text{for } m \geq 1 \quad \text{.....(5)}$$

Fig. 2. Linear thermal expansion of three carbon steels as a function of temperature.

$$Y = \frac{4\varepsilon_s \sigma T_0^3 l}{\alpha \rho C_p} \quad \text{.....(6)}$$

where $T_M = Q/C$ is the maximum temperature rise of the rear surface of the specimen with no heat loss, Q is the total energy absorbed by the specimen, C is the heat capacity of the specimen, σ is the Stefan–Boltzmann constant, T_0 is the steady-state temperature of specimen, l is the thickness of specimen, ρ is the density of specimen, C_p is the specific heat capacity, and ε_s is the emissivity of the surface of specimen. Thermal diffusivity value, α , can be obtained by fitting Eq. (1) to the measured temperature response with a least square method. The values of Y and T_M are experimentally obtained from the cooling part of the measured temperature response curve. It may be worthy of note that the values of ρ , C_p and ε_s are not required to determine the thermal diffusivity in this data processing, as already described in detail by Cezarian *et al.*⁴⁾

3. Results and Discussion

3.1. Linear Thermal Expansion of Steels

The linear thermal expansion values of six steels were measured in the temperature range from 603 to 1 680 K. The percentage linear thermal expansion of three carbon steels is shown in Fig. 2 as a function of temperature. The recommended values for percentage linear thermal expansion of iron⁵⁾ were also illustrated by dashed-and-dotted line in Fig. 2. The percentage linear thermal expansion of ultra low carbon steel decreases in the temperature range from 1 175 to 1 180 K. Similar behavior is detected in the temperature range from 990 to 1 160 K for low carbon steel and that from 990 to 1 115 K for medium carbon steel, respectively. Such decrease corresponds to the phase transformation from ferrite (bcc) phase to austenite (fcc) phase. The amount of the change of the elongation due to the transformation is less than that of the literature value of iron as shown in Fig. 2. A part of the reason of this difference may be explained by a difference of the chemical composition between the specimens used in this work and the

Fig. 3. Linear thermal expansion of three special steels as a function of temperature.

specimen of iron in the literature as well as the experimental conditions. However, there are no obvious explanations for the differences between them. On the other hand, the percentage linear thermal expansion of ultra low carbon steel increases in the temperature close to 1660 K. This variation is attributed to the phase transformation from austenite (fcc) to δ -ferrite (bcc) and they are coincident well with the values given in the Fe–C phase diagram.⁶⁾

The percentage linear thermal expansion of three special steels is shown in Fig. 3 as a function of temperature. The variation, corresponding to the phase transformation from ferrite (bcc) to austenite (fcc), is detected in the temperature range from 900 to 975 K for the 9% nickel steel and from 1070 to 1115 K for the 13% chromium steel, respectively. However, only the monotonic increase is observed in the percentage linear thermal expansion of the 1.2% silicon steel with increasing temperature, because only ferrite (bcc) phase can be available in the 1.2% silicon steel as confirmed by the Fe–Si phase diagram.⁷⁾ The percentage linear thermal expansion, $100\Delta L/L_{RT}$,* of six steels was fitted using a polynomial regression (fourth term), similar to the previous cases^{8,9)} for further analysis of thermal diffusivity as a function of T in K. The uncertainty of the linear thermal expansion of the six steels is $\pm 2\%$, based on the previous results for silicon using the same apparatus.¹⁰⁾ The regression coefficients obtained are listed in Table 2.

3.2. Estimation of the Decrease in the Specimen Thickness

After the thermal diffusivity measurement close to solidus of the specimens, the reduction in both weight and thickness was clearly observed for all steel specimens. The changes are summarized in Table 3. The variation detected in thickness may be mainly due to evaporation of oxides formed on the specimen surface because the relatively low melting point is cited for these oxides. On the other hand, the change in diameter of the steel specimen was not observed before and after the measurement. In order to deter-

Table 2. Percentage linear thermal expansion values (unit: %) of ultra low carbon steel (ULCS), low carbon steel (LCS), medium carbon steel (MCS), 1.2% silicon steel (1.2% Si), 9% nickel steel (9% Ni) and 13% chromium steel (13% Cr) fitted using a polynomial regression. The uncertainty of linear thermal expansion of six steels is suggested to be $\pm 2\%$, based on the previous work on silicon using the same apparatus.¹⁰⁾

Steel	Temperature range (K)	Regression $100\Delta L/L_{RT}=a_0+a_1T+a_2T^2+a_3T^3+a_4T^4$				
		a_0	a_1	a_2	a_3	a_4
ULCS	$603 \leq T \leq 1170$	0.347	-0.00252	7.279×10^{-6}	-5.673×10^{-9}	1.605×10^{-12}
	$1190 \leq T \leq 1640$	-1.068	0.00204	-2.483×10^{-7}	1.629×10^{-10}	-1.875×10^{-15}
LCS	$603 \leq T \leq 990$	-0.215	7.709×10^{-5}	2.685×10^{-6}	-2.228×10^{-9}	7.271×10^{-13}
	$1180 \leq T \leq 1670$	-8.520	0.02160	-2.027×10^{-5}	9.324×10^{-9}	-1.589×10^{-12}
MCS	$603 \leq T \leq 980$	-0.070	-0.00122	6.126×10^{-6}	-5.877×10^{-9}	2.063×10^{-12}
	$1150 \leq T \leq 1680$	-6.485	0.01601	-1.451×10^{-5}	6.709×10^{-9}	-1.155×10^{-12}
1.2 % Si	$603 \leq T \leq 1680$	-0.771	0.00267	-1.479×10^{-6}	6.820×10^{-10}	-5.656×10^{-14}
9 % Ni	$603 \leq T \leq 870$	-9.743	0.05222	-1.033×10^{-4}	9.290×10^{-8}	3.123×10^{-11}
	$990 \leq T \leq 1630$	-1.531	0.00223	-2.579×10^{-7}	1.602×10^{-10}	-2.229×10^{-14}
13 % Cr	$603 \leq T \leq 980$	-1.229	0.00518	-6.602×10^{-6}	4.748×10^{-9}	-1.212×10^{-12}
	$1190 \leq T \leq 1630$	28.216	-0.08823	1.018×10^{-4}	-5.059×10^{-8}	9.367×10^{-12}

Table 3. Changes of weight and thickness for ultra low carbon steel (ULCS), low carbon steel (LCS), medium carbon steel (MCS), 1.2% silicon steel (1.2% Si), 9% nickel steel (9% Ni) and 13% chromium steel (13% Cr).

	ULCS	LCS	MCS	1.2 % Si	9 % Ni	13 % Cr
weight (g)						
before meas.	0.634	0.642	0.631	0.624	0.636	0.624
after meas.	0.576	0.589	0.577	0.568	0.589	0.562
change in weight	-0.058	-0.053	-0.054	-0.056	-0.047	-0.062
thickness (mm)						
before meas.	1.030	1.048	1.035	1.030	1.032	1.032
after meas.	0.950	0.970	0.955	0.945	0.975	0.945
change in thickness	-0.080	-0.078	-0.080	-0.085	-0.057	-0.087

mine thermal diffusivity, an accurate value of the specimen thickness is essentially required at desired temperature.

In order to estimate the thickness change at elevated temperature, the following procedure was employed. After heating for 30 min at 1572, 1622 and 1676 K, the steel specimens were quenched by furnace cooling. The quenching rate was the order of 100 K/s at 1573 K. The thickness of the quenched specimens was measured at room temperature using a micrometer. The true decrease in thickness of steel specimen, β (mm), in the vertical axis at the elevated temperature is introduced to estimate the thickness change, and this parameter can be given by Eq. (7);

$$\beta = l_{\text{loss}} + l_{\text{loss}} \times \frac{\Delta L}{L_{RT}} \quad \text{.....(7)}$$

where l_{loss} is the decrease of thickness of the as-quenched specimen. The value of β of three carbon steels is shown in Fig. 4 and that of special steels is given in Fig. 5, as a function of temperature difference defined by $T_s - T$, where T_s and T are solidus¹¹⁾ and measured temperature. The specimen thickness drastically decreases with increasing temperature so as to be the temperature difference, $T_s - T$, less than 200 K. When the value of, $T_s - T$, is 100 K, the decrease in thickness is 0.06 mm for the 9% nickel steel and 0.085 mm for other steels.

* L_{RT} is the length of a specimen for measuring the linear thermal expansion coefficient at room temperature, and ΔL is the elongation measured by the dilatometer. The expressions are not given in the two-phase region because no measurement of thermal diffusivity is made in that region.

Fig. 4. Thickness change of three carbon steels as a function of temperature.

Fig. 5. Thickness change of 1.2% silicon, 9% nickel, and 13% chromium steels.

3.3. Measurements of Thermal Diffusivity

From the results of the linear thermal expansion coefficient and the change in thickness of steel specimens, the specimen thickness, l_T , at elevated temperature can be given by Eq. (8);

$$l_T = \left(l_{RT} + l_{RT} \times \frac{\Delta L}{L_{RT}} \right) - \beta \quad \text{.....(8)}$$

where l_{RT} is the specimen thickness at room temperature. By using the relation of Eq. (19), the correction for specimen thickness is made for determining the thermal diffusivity value at temperature near solidus from measured temperature response curve. The measured temperature response curve is shown in Fig. 6 using the result of ultra low carbon steel at 773 K as an example. The temperature response of the bottom surface of the specimen instantaneously increases after laser irradiation, and shows the maximum value, then decreases due to the radiative heat loss from the surface of the specimen. This temperature response curve is

Fig. 6. Experimental and fitted temperature response curves of ultra low carbon steel at 773 K.

Fig. 7. Thermal diffusivity of three carbon steels as a function of temperature.

fitted by Eq. (1), then the thermal diffusivity value can be obtained. Solid line in Fig. 6 is denoted by the fitted temperature response curve which reproduces well the measured data.

The thermal diffusivity values obtained in the temperature range from room temperature to 1676 K are shown in Fig. 7 for ultra low carbon, low carbon, and medium carbon steels. The recommended values for thermal diffusivity of Armco iron are available in the temperature range between 700 K and 1400 K. The recommended values¹⁾ are also shown in Fig. 7. The measured values show the same tendency and good agreement with the literature values. The results are summarized as follows:

(1) The thermal diffusivity values of medium carbon steel, its carbon concentration is relatively high, is found to be smaller than those of ultra low and low carbon steels in the temperature range below ferrite-austenite A_1 transformation point (1013 K).⁶⁾

Fig. 8. Thermal diffusivity of three special steels as a function of temperature.

(2) The thermal diffusivity values of three carbon steels decreases with increasing temperature below ferrite–austenite A_1 transformation point.

(3) The thermal diffusivity values of three carbon steels slightly increase with increasing temperature above ferrite–austenite A_1 transformation point.

On the other hand, the thermal diffusivity values for special steels are shown in Fig. 8. The results are summarized as follows:

(1) The thermal diffusivity values of special steels decrease with increasing temperature up to 1 000 K.

(2) The thermal diffusivity values of special steels slightly increase when temperature is higher than 1 000 K. It may be noted that similar temperature dependence of thermal diffusivity are also found in several alloy specimens.¹²⁾

(3) The thermal diffusivity values of special steels are smaller than those of carbon steels in the temperature range from room temperature to 1 000 K.

4. Summary

In this work, the thermal diffusivity values of six steels were successfully determined by estimating the specimen thickness accurately at elevated temperature near solidus. Thermal diffusivity values of six steels are summarized in Table 4. Measured thermal diffusivity values, α , of six steels in the temperature range of austenite phase were fitted using a linear regression. The results in the unit of $\text{mm}^2 \text{s}^{-1}$ are given below (T in K) for further convenience. The regression coefficients are listed in Table 5.

The uncertainty of thermal diffusivity of six steels below 1 375 K is $\pm 1.6\%$. The uncertainty of thermal diffusivity of six steels above 1 572 K increases to $\pm 5.8\%$ because the uncertainty of β . It would be interesting to extend the present results to computer simulation for some steel making process, especially for designing the appropriate hot rolling and casting process with sufficient reliability.

Table 4. Thermal diffusivity values (unit: $\text{mm}^2 \text{s}^{-1}$) of ultra low carbon steel (ULCS), low carbon steel (LCS), medium carbon steel (MCS), 1.2% silicon steel (1.2% Si), 9% nickel steel (9% Ni) and 13% chromium steel (13% Cr).

Temperature (K)	ULCS	LCS	MCS	1.2 % Si	9 % Ni	13 % Cr
room temperature	19.9	19.8	12.2	9.07	7.96	7.24
773	9.62	9.60	7.89	7.18	6.90	5.61
973	5.53	5.70	5.03	4.81	4.65	3.93
1176	–	5.84	5.89	4.94	5.79	5.74
1273	6.14	6.21	5.99	5.38	6.08	5.84
1375	6.44	6.37	6.18	5.69	6.11	5.72
1572	6.60	6.84	6.46	5.56	6.25	6.00
1622	6.59	6.81	6.19	5.35	6.20	6.06
1676	6.52	6.88	6.02	5.69	5.95	5.71

Table 5. Thermal diffusivity values (unit: $\text{mm}^2 \text{s}^{-1}$) of ultra low carbon steel (ULCS), low carbon (LCS), medium carbon steel (MCS), 1.2% silicon steel (1.2% Si), 9% nickel steel (9% Ni) and 13% chromium steel (13% Cr) in the temperature range of austenite phase fitted using a linear regression. The uncertainty of thermal diffusivity of six steels below 1 375 K is $\pm 1.6\%$. The uncertainty of thermal diffusivity of six steels above 1 572 K is $\pm 5.8\%$.

Steel	Temperature range (K)	Regression $\alpha = b_0 + b_1 T$	
		b_0	b_1
ULCS	$1273 \leq T \leq 1622$	3.95	1.76×10^{-3}
LCS	$1176 \leq T \leq 1676$	3.20	2.30×10^{-3}
MCS	$1176 \leq T \leq 1676$	4.80	9.50×10^{-4}
1.2 % Si	$1176 \leq T \leq 1676$	2.71	2.03×10^{-3}
9 % Ni	$1176 \leq T \leq 1676$	4.80	9.28×10^{-4}
13 % Cr	$1176 \leq T \leq 1676$	5.56	1.66×10^{-4}

Acknowledgements

The authors wish to express their gratitude to Prof. H. Ohtani, Dr. K. Yoshimi and Dr. T. Haraguchi, Center for Interdisciplinary Research Tohoku University, for their help on the thermal expansion measurements.

Nomenclature

- T_r : Temperature rise of the rear surface of the specimen (K)
- T_M : Maximum temperature rise of the rear surface of the specimen with no heat loss (K)
- Q : Total energy absorbed by the specimen (J)
- C : Heat capacity of specimen (J K^{-1})
- σ : Stefan–Boltzmann constant ($\text{W mm}^{-2} \text{K}^{-4}$)
- T_0 : Steady-state temperature of specimen (K)
- l : Thickness of specimen at T_0 (mm)
- ρ : Density of the specimen (g mm^{-3})
- C_p : Specific heat capacity of specimen ($\text{J g}^{-1} \text{K}^{-1}$)
- ϵ_s : Emissivity of the surface of specimen
- α : Thermal diffusivity ($\text{mm}^2 \text{s}^{-1}$)
- β : Decrease in thickness of specimen for the measurement of thermal diffusivity (mm)
- l_T : Estimated thickness of specimen at elevated temperature of T (mm)
- l_{RT} : Thickness of specimen at room temperature for the measurement of thermal diffusivity (mm)
- L_{RT} : Length of specimen at room temperature for the measurement of linear thermal expansion (mm)
- ΔL : Elongation of specimen at each measurement temperature (mm)

$100\Delta L/L_{RT}$: Percentage linear thermal expansion

REFERENCES

- 1) Y. S. Touloukin, R. W. Powell, C. Y. Ho and M. C. Nicolaou: Thermophysical Properties of Matter., Vol. 10, IFI/PLENUM, New York, (1973), 82.
- 2) Y. S. Touloukin, R. W. Powell, C. Y. Ho and M. C. Nicolaou: Thermophysical Properties of Matter., Vol. 12, IFI/PLENUM, New York, (1973), 868.
- 3) W. J. Parker, R. J. Jenkins, C. P. Butler and G. L. Abbott: *J. Appl. Phys.*, **32** (1961), 1679.
- 4) A. Cezairliyan, T. Baba and R. Taylor: *Int. J. Thermophys.*, **15** (1994), 317.
- 5) Y. S. Touloukin, R. W. Powell, C. Y. Ho and M. C. Nicolaou: Thermophysical Properties of Matter., Vol. 12, IFI/PLENUM, New York, (1973), 157.
- 6) T. B. Massalski, H. Okamoto, P. R. Subramanian and L. Kacprzak: Binary Alloy Phase Diagrams, 2nd Ed., Vol. 1, ASM International, Materials Park, OH, (1990), 842.
- 7) T. B. Massalski, H. Okamoto, P. R. Subramanian and L. Kacprzak: Binary Alloy Phase Diagrams, 2nd Ed., Vol. 2, ASM International, Materials Park, OH, (1990), 1771.
- 8) M. D. Mathews, B. R. Ambekar and A. K. Tyagi: *J. Nucl. Mater.*, **288** (2001), 83.
- 9) A. K. Tyagi, M. D. Mathews and R. Ramachandran: *J. Nucl. Mater.*, **294** (2001), 198.
- 10) N. Yamada, R. Abe and M. Okaji: *Meas. Sci. Technol.*, **12** (2001), 2121.
- 11) Handbook of Iron and Steel (Tekkobenran), ed. by ISIJ, Maruzen, Tokyo, (1981), 205.
- 12) Y. S. Touloukin, R. W. Powell, C. Y. Ho and M. C. Nicolaou: Thermophysical Properties of Matter., Vol. 10, IFI/PLENUM, New York, (1973), 337.