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Validation of Calculated Thermal Parameters with Experimental Results in SOFCs

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Abstract

Eleven steel grades were designed to be used as metallic interconnects for Solid Oxide Fuel Cells (SOFC). Low carbon, high chromium steel with different additives of niobium, vanadium, aluminum, molybdenum, silicon, manganese and titanium were produced. Phase transformation temperatures; eutectoid temperature (Ac1) and temperature at which transformation of ferrite to austenite is completed during heating (Ac3) were measured by L75-76 dilatometer. The influence of the alloying elements on transformation temperatures was analyzed using MATLab. Considering the interaction between different alloying elements two equations for predicting Ac1 & Ac3 were obtained. The obtained Ac1 & Ac3 by these equations showed more compatibility than that obtained by traditional ones. In addition, the coefficients of thermal expansion of these steel grades were detected. The influences of chemical composition and temperature on the thermal expansion coefficient were analyzed; the obtained equations were verified to certain extent by using several kinds of steels. The predicted values were in good accordance with the experimental results which proof the validation of calculation model.

Keywords

Transformation Temperature, Thermal Expansion Coefficient, SOFCs Steel, Matlab

1. Introduction

Recent research results have enabled to decrease the operating temperature of the Solid Oxide Fuel Cells (SOFCs) from 1000˚C to 800˚C [1]. This progress has been made by reducing the thickness of the electrolyte [2] and improving the cathode electrolyte interface reaction (*i.e.* Triple Phases Boundaries (TPB) to Internal Diffusion (ID) mechanism) [3]. The lower operating temperature authorises metallic alloys as possible candidates for interconnects [4]. Solid oxide fuel cells (SOFC) are environmental friendly energy conversion device with high efficiency and prolonged-ranging fuel utilization [5]. The thermal expansion coefficient (TEC) of interconnects should be around 10 - 13 × 10^{-6} K^{-1} [6] [7]. It was reported that the rare earth elements and their oxides can be used to decrease the oxidation rate [8] [9].

Metallic materials have higher electrical and thermal conductivities, are easier to fabricate, and, in general, have lower cost compared to the ceramic interconnects [4] [10]. Chromium is the most important element because of the formation of chromia as protective and semiconducting layer. The presence of other elements could improve the characteristics of this layer, limiting the growth rate and the acceptable area-specific resistance (ASR), reducing the poisoning of the electrodes due to the oxidation gaseous species (CrO_3 or $CrO_2(OH)_2$) at temperatures close to 1000˚C and higher [11] [12], but also observed at lower temperatures due to the severe operation conditions, such as the presence of water vapor [11] [12] [13] [14] [15]. The formation of a protective, single-phase chromium layer requires chromium content of approximately 17% - 20% [10] [16]-[21], depending on the temperature, surface treatment and minor alloying additions. Mn and Ti are used in a few tenths of the percent to improve the oxidation resistance. Mn tends to form a Cr-Mn spinel on the external surface layer to decrease the formation of volatile Cr species [10] [11] [17] [22] [23] [24].

Although the influences of chemical composition on transformation temperatures have been studied since the 1960s and several equations suitable for different situations were deduced by analyzing the corresponding data of hundreds types of steels [25] [26] [27], these classic equations were not high precise and not effective, as these analyses were too general and many types of steels were involved. Furthermore, as these analyses were mainly multiple linear regressions, the interactions of the alloying elements were seldom considered. In view of these facts, the phase transformation temperatures of SOFCs steel were studied systematically in this research. Two equations were obtained to predict Ac1 and Ac3 considering the effect of chemical compositions. The predicted Ac1 and Ac3 by designed equations are compared with the traditional ones.

In the case of SOFCs steel, small thermal expansion is required because higher coefficients always mean higher stresses during the periodic process of heating and cooling. Generally, coefficients of thermal expansion of steel will increase along with the increase in the total content of the alloying elements. The influences of the interactions of the elements on the coefficient are more complicated. Therefore, the thermal expansion coefficients of the samples at annealed state are also measured. Model has been established to predict the thermal expansion coefficient as a function of chemical composition and temperature. The novelty of this work; the effect of the interaction combination among different alloying elements and/or temperature on Ac1, Ac3 and thermal expansion coefficient was taken into consideration.

2. Experimental

Eleven developed ferritic stainless steel (SOFCs) with different refractory alloying elements additives were melted in induction furnace of capacity 10 kg and cast in sand mold. Complete chemical analysis has been carried out for all cast steels. The cast steels were normalized at 1000˚C for 4 hours, followed by open radial forging. Ingots with square diameter 65 mm were hot forged to about 35 mm square. The steel were reheated up to 1200˚C and hold for 2 hours before start forging. Starting forging temperature was 1150˚C while forging process was ended at temperatures 950˚C.

Thermal expansion measurements were carried out with L75-76 dilatometer. The specimens were prepared by machining from each steels to form a rectangular shape with the dimensions (3 × 3 × 30 mm) and polished through 600 grit prior to testing. Ac1 & Ac3 are estimated from the expansion curve against the temperature. The change in coefficient of thermal expansion was recorded during heating of the sample from room temperature to 1000oC and cooling from 1000 to 500oC. Two square matrices—of 10th degree—were designed as function of alloying elements and measured Ac1 or Ac3. Also, High order square matrix was designed between the alloying elements, thermal expansion coefficient at each temperature. MATLab was used to solve these higher order matrices to get Ac1, Ac3 and thermal expansion coefficient as function in alloying elements (for Ac1 and Ac3) and temperature (Thermal expansion coefficient).

3. Result and Discussion

The chemical composition of developed SOFCs ferritic steel grades are listed in Table 1. With L75-76 dilatometer, the transformation temperatures Ac1 & Ac3 of the investigated steels were measured, and the results are shown in Table 2.

The data listed in Table 1 and Table 2 was analyzed using MATLab. The relationships between the chemical composition and phase transformation temperatures, Ac1 and Ac3, were studied, where two corresponding equations were deduced as follows:

$$\begin{aligned} \mathrm{Ac1} = {} & 45791.55*[\mathrm{C\%}] - 150.551*[\mathrm{Si\%}] + 1385.216*[\mathrm{Mn\%}] - 27.208*[\mathrm{Cr\%}] \\ & + 495.9697*[\mathrm{Mo\%}] - 700.922*[\mathrm{Nb\%}] + 39115.62*[\mathrm{V\%}] \\ & - 982.165*[\mathrm{Mn\%}]^2 - 1606.56*[\mathrm{Cr\%}]*[\mathrm{C\%}] \\ & - 7448.37*[(\mathrm{Nb}+\mathrm{V}+\mathrm{Mo})\%]*[\mathrm{C\%}] \end{aligned} \tag{1}$$

$$\begin{aligned} \mathrm{Ac3} = {} & 43732.4*[\mathrm{C\%}] - 161.931*[\mathrm{Si\%}] + 1294.837*[\mathrm{Mn\%}] - 25.4274*[\mathrm{Cr\%}] \\ & + 484.8949*[\mathrm{Mo\%}] - 606.577*[\mathrm{Nb\%}] + 38400.17*[\mathrm{V\%}] \\ & - 923.807*[\mathrm{Mn\%}]^2 - 1509.54*[\mathrm{Cr\%}]*[\mathrm{C\%}] \\ & - 7417.06*[(\mathrm{Nb}+\mathrm{V}+\mathrm{Mo})\%]*[\mathrm{C\%}] \end{aligned} \tag{2}$$

From Equations (1) & (2), it can be seen that, the carbon has a positive effect

Table 1. Chemical composition of $SOFC_S$ steel grades, wt%.

Heat No.	C	Si	Mn	Cr	Mo	Al	Nb	Ti	V
1	0.060	2.00	0.62	33.01	0.052	0.0001	0.0040	0.0037	0.043
2	0.079	0.35	1.55	25.16	0.060	0.0005	0.0029	0.0031	0.029
3	0.177	0.37	0.08	23.43	1.150	0.0167	0.0051	0.0032	0.018
4	0.065	1.13	0.08	22.11	0.049	0.0072	0.0057	0.0029	0.021
5	0.067	0.29	0.14	25.94	0.050	0.0256	0.0020	0.0781	0.024
6	0.101	2.20	0.85	23.30	0.906	0.0212	0.6190	0.0602	0.036
7	0.055	1.05	0.13	25.71	0.909	0.0105	0.0303	0.0083	0.024
8	0.078	1.25	0.83	28.81	0.043	0.0105	0.0076	0.0923	0.028
9	0.063	2.64	0.27	30.46	0.052	0.6550	0.0004	0.0107	0.042
10	0.051	0.43	0.17	27.13	0.054	1.5700	0.0044	0.0067	0.026
11	0.076	0.38	1.41	25.10	1.160	0.0217	0.0026	0.0032	0.025

Table 2. Measured transformation temperatures of the investigated steels, ˚C.

Heat No.	Ac1 (˚C)	Ac3 (˚C)
1	520	720
2	560	650
3	500	720
4	680	840
5	530	640
6	500	610
7	480	780
8	480	630
9	460	610
10	480	720
11	480	700

on increasing both Ac1 & Ac3. But in presence of Cr, Nb, V and Mo; carbon will cause decreasing in both Ac1 & Ac3. On the other hand, Si, Cr and Nb have a tendency to lower Ac1 & Ac3 but Mo and V have a significant effect in increasing both Ac1 & Ac3. Meanwhile Mn has a special effect as its effect is the sum of two opposite effect; the first is positive one which related to the manganese content of the metal to the power one while the second effect related to the manganese content of the metal to the power two. This means that Mn might increase or decrease Ac1 & Ac3 depending on its content in steel. Generally, within the range of this study, V and Cr are the main alloying elements that affected phase transformation temperatures, whereas, Mn, C, Mo, and Si were the less important ones.

The coefficients of thermal expansion (α) of annealed SOFCs steel grades at different temperatures are listed in **Table 3**. On the basis of the data listed in **Table 1** and **Table 3**, the influences of chemical composition and temperature on the thermal expansion coefficient of different steel grades were studied, and an equation was deduced to predict the thermal expansion coefficient as a function of alloying elements and temperature as given in Equation (3)

$$\begin{aligned}&\text{Thermal Expansion}(\alpha)(*10^{-6})\\&=240*[\text{C}\%]+1.99*[\text{Si}\%]+0.489*[\text{Mn}\%]+0.249*[\text{Cr}\%]+5.41*[\text{Mo}\%]\\&\quad-3.63*[\text{Al}\%]-7.13*[\text{Cr}\%]*[\text{C}\%]-62.9*[\text{Mo}\%]*[\text{C}\%]\\&\quad-66.3*[(\text{Nb}+\text{Ti}+\text{V})\%]*[\text{C}\%]+0.004416*\text{T}-3.532468\end{aligned} \quad (3)$$

From Equation (3), it can be noticed that: Mn, Si, Mo, Cr and C have a tendency to increase α, but the increasing effect is restrained by the presence of Al and carbides of Mn, Nb, Mo, Nb, Ti, V . At the same time the temperature has small effect on increasing coefficient of thermal expansion.

In this section, the calculated thermal expansion by Equation (3) is compared by that obtained by applying the traditional Equations (4)-(5), which were given by Andrews [20] [21] and modern Equations (6)-(7) given by XIE Hao-jie [28]:

$$\begin{aligned}\text{Ac1}=723-10.7*[\text{Mn}\%]-16.9*[\text{Ni}\%]+29*[\text{Si}]\\+16.9*[\text{Cr}]+290*[\text{As}\%]+6.38*[\text{W}\%]\end{aligned} \quad (4)$$

$$\begin{aligned}\text{Ac3}=910-203*[\text{C}\%]^{1/2}-15.2*[\text{Ni}\%]+44.7*[\text{Si}\%]\\+104*[\text{V}\%]+31.5*[\text{Mo}\%]+13.1*[\text{W}\%]\end{aligned} \quad (5)$$

$$\begin{aligned}\text{Ac1}=36.57605*[\text{Mn}\%]-6.279322*[\text{C}\%]*[\text{Cr}\%]\\-74.38445*[\text{C}\%]*[\text{V}\%]-51.62571*[\text{Mn}\%]^2+858.9063\end{aligned} \quad (6)$$

$$\begin{aligned}\text{Ac3}=777.1057+52.85982*[\text{C}\%]*[\text{Cr}\%]-10.23115*[\text{Cr}\%]*[\text{Mo}\%]\\+72.39112*[\text{V}\%]2+26.68782*[\text{Mo}\%]*[\text{V}\%]\end{aligned} \quad (7)$$

Table 3. Experimental result of thermal expansion coefficients of the steels (X 10^{-6}/˚C).

Heat No.	30˚C	100˚C	200˚C	300˚C	400˚C	500˚C	600˚C	700˚C	800˚C
1	9.2	10.4	10.93	11.37	11.55	11.65	11.66	12.14	12.66
2	9.0	10.57	10.99	11.45	11.66	11.73	11.65	11.98	12.33
3	8.99	10.62	11.25	11.69	11.85	11.81	11.62	11.88	12.55
4	8.62	10.73	11.42	11.91	12.21	12.44	12.62	12.83	13.04
5	8.81	10.84	11.83	12.38	12.57	12.64	12.51	12.83	13.25
6	8.67	10.34	10.97	11.46	11.67	11.73	11.67	11.97	12.34
7	8.81	10.33	10.82	11.30	11.53	11.62	11.52	11.81	12.08
8	8.79	10.32	10.77	11.23	11.40	11.49	11.34	11.65	12.09
9	9.19	10.65	11.24	11.71	11.86	11.74	11.60	11.95	11.88
10	8.70	10.39	11.07	11.57	11.76	11.77	11.62	11.92	12.56
11	8.75	10.32	10.80	11.26	11.47	11.60	11.52	11.72	12.82

It should be mentioned that Equation (4) and Equation (5) were obtained by considering the influence of every single alloying element on the corresponding phase transformation, temperature only, whereas, Equation (6) and Equation (7) were deduced with consideration of the interaction of the alloying elements.

On the basis of the chemical composition of different steel grades as listed in Table 1, the phase transformation temperatures (Ac1 & Ac3) of the steels were calculated using Equations (1)-(2) and Equations (4)-(7). The calculated values of both Ac1 and Ac3 are listed in Table 4 and represented in Figures 1-2 respectively.

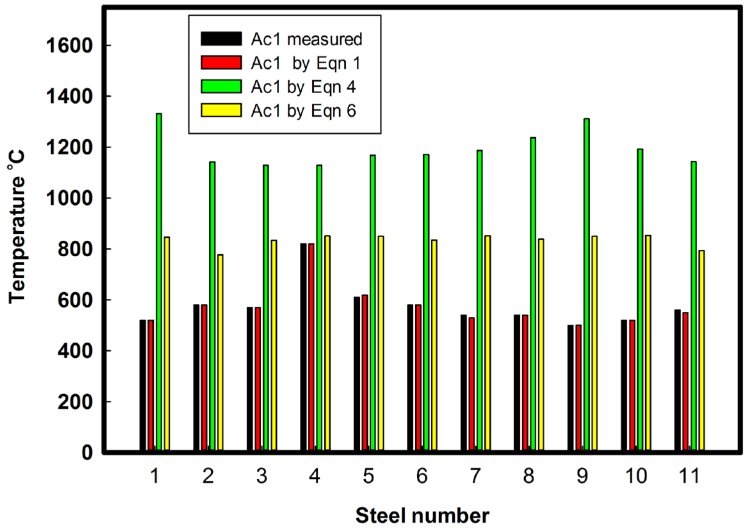

Figure 1. Variation between the measured Ac1 values with the estimated values of the authors (Equation (1)), Andrews [20] [21] (Equation (4)) and XIE Hao-jie [28] (Equation (6)).

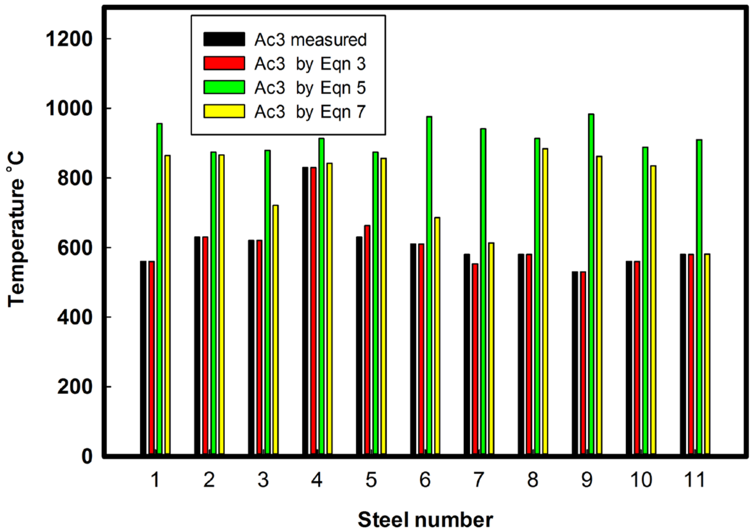

Figure 2. Variation between the measured Ac3 values with the estimated values of the authors (Equation (2)), Andrews [20] [21] (Equation (5)) and XIE Hao-jie [28] (Equation (7)).

Table 4. Validity of calculated phase transformation temperatures with experimental results.

Heat No.	Ac1				Ac3			
	Measured	Equation (1)	Equation (4)	Equation (6)	Measured	Equation (2)	Equation (5)	Equation (7)
1	520	520.0	1332	846	560	560.0	956	864
2	580	580.0	1142	777	630	630.0	874	866
3	570	570.0	1129	834	620	620.0	879	721
4	820	820.0	1129	851	830	830.0	913	842
5	610	618.1	1168	850	630	662.7	874	856
6	580	580.0	1171	835	610	610.0	976	686
7	540	529.1	1187	852	580	553.3	941	613
8	540	540.0	1237	838	580	580.0	913	884
9	500	500.3	1311	850	530	530.2	983	862
10	520	520.0	1192	853	560	560.0	888	835
11	560	550.0	1143	794	580	580.0	910	581

It can be noticed that as illustrated in **Figures 1-2**, the values obtained by Equations (6)-(7) are more close to the measured one than that obtained by Equations (4)-(5). This is because Equations (6)-(7) take into consideration the interaction between the elements. On the other hand, the values calculated by the current work, Equations (1)-(2), are more consistent with the experimental values than that obtained by Equations (4)-(7).

The measured thermal expansion coefficients of the investigated annealed SOFCs steel grades were compared with that estimated by Equation (3) which designed by current work and Equation (8) which designed by XIE Hao-jie [28]. The measured and estimated values of thermal expansion coefficients are given in **Table 5**.

$$\begin{aligned}&\text{Thermal Expansion Coefficient}(\alpha)\\&=-0.530592*[\text{Si\%}]*[\text{V\%}]+0.696172*[\text{Mn\%}]*[\text{Mo\%}]\\&\quad-0.173824*[\text{Cr\%}]*[\text{Mo\%}]+0.001401*[\text{Cr\%}]*\text{T}\\&\quad+0.001966*[\text{V\%}]*\text{T}-0.000005\text{T}^2+11.8656\end{aligned} \tag{8}$$

It can be noticed that, for the steels tested in this experiment, the predicted values by current work equation, Equation (3), are more consistent with the experimental values than that obtained by Equation (8). This may be due to Equation (3) take into consideration the effect of the carbon and Aluminum.

4. Conclusion

It is difficult to build up equation to predict Ac1, Ac3 and thermal expansion coefficient for a wide range of chemical compositions of different steel grades. But it is possible to build up Equations to predict Ac1, Ac3 and coefficient of thermal expansion for limited chemical composition range for certain steel

Table 5. Thermal expansion coefficients of the steels (X 10^{-6}/˚C), measured, expected by Equations (3) & (8).

Heat No.		30˚C	100˚C	200˚C	300˚C	400˚C	500˚C	600˚C	700˚C	800˚C
	Measured	9.2	10.4	10.93	11.37	11.55	11.65	11.66	12.14	12.66
1	Equation (3)	9.26	9.57	10.01	10.45	10.89	11.34	11.78	12.22	12.66
	Equation (8)	12.93	16.13	20.61	24.99	29.28	33.46	37.54	41.53	45.41
	Measured	9.0	10.57	10.99	11.45	11.66	11.73	11.65	11.98	12.33
2	Equation (3)	8.94	9.24	9.69	10.13	10.57	11.01	11.45	11.89	12.34
	Equation (8)	12.72	15.14	18.52	21.81	24.99	28.07	31.05	33.93	36.71
	Measured	8.99	10.62	11.25	11.69	11.85	11.81	11.62	11.88	12.55
3	Equation (3)	9.17	9.48	9.92	10.36	10.80	11.24	11.68	12.12	12.57
	Equation (8)	8.23	10.48	13.62	16.65	19.59	22.42	25.16	27.80	30.33
	Measured	8.62	10.73	11.42	11.91	12.21	12.44	12.62	12.83	13.04
4	Equation (3)	9.64	9.95	10.39	10.83	11.27	11.72	12.16	12.60	13.04
	Equation (8)	12.59	14.72	17.67	20.52	23.27	25.93	28.48	30.93	33.28
	Measured	8.81	10.84	11.83	12.38	12.57	12.64	12.51	12.83	13.25
5	Equation (3)	6.89	7.20	7.64	8.08	8.53	8.97	9.41	9.85	10.29
	Equation (8)	12.73	15.23	18.72	22.11	25.40	28.59	31.67	34.66	37.55
	Measured	8.67	10.34	10.97	11.46	11.67	11.73	11.67	11.97	12.34
6	Equation (3)	8.94	9.25	9.69	10.13	10.57	11.02	11.46	11.90	12.34
	Equation (8)	9.67	11.91	15.04	18.06	20.98	23.80	26.52	29.14	31.66
	Measured	8.81	10.33	10.82	11.30	11.53	11.62	11.52	11.81	12.08
7	Equation (3)	9.78	10.09	10.53	10.97	11.41	11.86	12.30	12.74	13.18
	Equation (8)	8.95	11.43	14.88	18.24	21.50	24.65	27.71	30.67	33.52
	Measured	8.79	10.32	10.77	11.23	11.40	11.49	11.34	11.65	12.09
8	Equation (3)	8.69	9.00	9.44	9.88	10.32	10.76	11.21	11.65	12.09
	Equation (8)	12.87	15.65	19.54	23.33	27.02	30.62	34.11	37.50	40.79
	Measured	9.19	10.65	11.24	11.71	11.86	11.74	11.60	11.95	11.88
9	Equation (3)	8.47	8.78	9.23	9.67	10.11	10.55	10.99	11.43	11.87
	Equation (8)	12.82	15.77	19.90	23.92	27.85	31.67	35.40	39.02	42.55
	Measured	8.70	10.39	11.07	11.57	11.76	11.77	11.62	11.92	12.56
10	Equation (3)	0.95	1.26	1.72	2.14	2.59	3.03	3.47	3.91	4.35
	Equation (8)	12.75	15.37	19.02	22.58	26.03	29.39	32.65	35.80	38.86
	Measured	8.75	10.32	10.80	11.26	11.47	11.60	11.52	11.72	12.82
11	Equation (3)	9.43	9.74	10.19	10.63	11.07	11.51	11.95	12.39	12.83
	Equation (8)	8.99	11.41	14.78	18.05	21.22	24.30	27.27	30.14	32.91

category. For SOFCs steel grades, within range of chemical compositions (0.0506% - 0.101% C, 0.354% - 2.2% Si, 0.0808% - 1.55% Mn, 25.1% - 33.01% Cr, 0.0427% - 1.1% Mo, 0.0001% - 1.5% Al, 0.0004% - 0.0923% Nb and 0.0177% - 0.0433% V) Ac1 and Ac3 Eqns. were deduced as a function in chemical composition. It could be concluded also that, coefficient of thermal expansion can be predicted as a function in chemical composition and temperature.

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