Research Article

Precipitation of σ Phase Using General Diffusion Equation with Comparison to Vitek Diffusion Model in Dissimilar Stainless Steels

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This study performs a precipitation examination of the σ phase using the general diffusion equation with comparison to the Vitek model in dissimilar stainless steels during multipass welding. Experimental results demonstrate that the diffusivities (D_{Cr}^{δ} , D_{Ni}^{δ} , and D_{Si}^{δ}) of Cr, Ni, and Si are higher in δ -ferrite than (D_{Cr}^{γ} , D_{Ni}^{γ} , and D_{Si}^{γ}) in the γ phase, and that they facilitate the precipitation of the σ phase in the third pass fusion zone. The Vitek diffusion equation can be modified as follows: $(\text{Diffusivity})_{\text{General}} = \text{Modified Constant} \times (\text{Diffusivity})_{\text{Vitek}}$.

1. Introduction

ASSs (austenitic stainless steels) are used widely in high-temperature conditions such as energy conversion systems. For instance, to ensure economy in central power systems, sections of boilers subjected to lower temperatures are constructed from FSS (ferritic stainless steel) for economic reasons [1]. However, ASS and FSS composites have acid, alkali, and high-temperature resistances, and so have many applications in a wide variety of industries, such as chemical, oil refining, artificial fiber, food, and medicine [2].

When stainless steels are heated to temperatures between 700°C and 1000°C for prolonged periods of time, several harmful second phases, such as σ , χ , and π , can precipitate [3–6]. While the σ phase was first observed in the Fe-Cr system, it has also been observed in Fe-Mo, Fe-V, and Cr-Mo-Ni alloy systems [7]. The crystallographic lattice of the σ phase is a tetragonal structure with 30 atoms per unit cell. The σ phase is the most important of these intermetallic phases due to its impact on the mechanical properties, corrosion resistance or weldability of stainless steels, as well as other properties [8–11].

Previous studies on the properties of stainless steels containing the σ phase have focused on microstructural observation and the mechanical property in the fusion zone

(FZ) of similar stainless steels [12–18]. Very few researchers have discussed the precipitation of the σ phase through the diffusion theory in the fusion zones (FZ) of dissimilar stainless steels. The precipitation behavior of the σ phase in fusion zones during the welding process is unclear for ferritic and austenitic stainless steels. Hsieh and Wu [19] first reported the precipitation of the σ phase using the Vitek diffusion model in the multipass fusion zone of dissimilar stainless steels.

Autogenous GTAW (gas tungsten arc welding) was used to manufacture dissimilar stainless steels for this present study. The precipitation behavior of the σ phase in the multipass fusion zone of dissimilar stainless steels taking place during the multipass GTAW process has been discussed via a calculation of the general diffusion equation with comparison to the Vitek diffusion model. The objectives were to delineate the precipitation mechanism of the σ phase in dissimilar stainless steels at various welding passes, as well as to contribute to the understanding of the precipitation behavior of the σ phase during the multipass GTAW process in dissimilar stainless steels.

2. Experimental Procedures

Two types of stainless steels, namely, 304 and 430, were employed in the experiment. Both are representatives of

TABLE 1: Chemical composition of the raw samples.

Material	Element (wt%)						
	Cr	Ni	Si	Mn	C	Mo	Fe
304 S.S.	19.2	10.9	1.0	2.0	0.08	1.22	Bal.
430 S.S.	18.3	—	0.75	1.0	0.12	1.18	Bal.

FIGURE 1: Manufacturing process of the dissimilar stainless steels. (a) Top view; (b) cross-section.

fully austenitic, that is, containing a small number of ferrite phases and fully ferritic microstructures, respectively. Their chemical compositions are listed in Table 1. The dimensions of the raw materials were 70 mm $\times$ 25 mm $\times$ 3 mm.

The composites of the dissimilar stainless steels were created using an automatic GTAW process. The appearance of the weld metal from the dissimilar stainless steels is shown in Figure 1. The multipass welding (304/430, 304-1, and 304-2) was performed without filler at a welding current of 100 A and an arc voltage of 11 V with a travel speed of 120 mm/min. A series of symbols for 304/430, 304-1, and 304-2 were defined as the first, second, and third pass welds, respectively.

Thin foils for observation with a transmission electron microscope (JEOL JEM-2010, HRTEM) were prepared from the third pass fusion zone. Specimens were thinned to 50 μ m by abrasion on SiC papers, and electropolished (Struers Tenupol-5, Twin Jet Electro-Polisher) with a twin-jet unit at 35 V in a mixture of 25% glycerol, 5% perchloric acid, and 75% ethanol at a temperature of -10°C . The method of selected area diffraction (SAD) was employed to identify the δ -ferrite, σ phase, and γ phase. Their chemical compositions were identified using an energy dispersive spectrometer (Oxford Link ISIS, EDS).

The peak temperatures and the endothermic and exothermic peaks were examined using a differential scanning calorimeter (NETZSCH DSC 404 C) in order to discuss the precipitation of $\delta \rightarrow \sigma + \gamma_2$. The specimens were heated from 600°C to 1300°C at a heating rate of $20^{\circ}\text{C}\cdot\text{sec}^{-1}$. Subsequently, the diffusion parameters were calculated using

TABLE 2: TEM-EDS analysis of the third pass fusion zone (304-2).

Welding region	Phase	Element (wt%)			
		Fe	Cr	Ni	Si
304-2	δ	60.26	30.11	1.08	5.10
	σ	55.63	35.58	2.04	3.32

the general diffusion equation to explain the phase transformation of $\delta \rightarrow \sigma$. These diffusion parameters included diffusivity (D) and the atomic jump frequencies of the solute atoms (Γ).

3. Results and Discussion

3.1. TEM Identification of the δ -Ferrite and σ Phase. Figures 2(a)–2(d) showed the TEM diffraction pattern of the δ -ferrite and σ phase in the third pass fusion zone (304-2). Lots of grains were found in the third pass fusion zone, as shown in Figure 2(a). Two phases can be observed clearly from a dark field image of Figure 2(b) and are identified as the δ -ferrite and σ phase using an identification of selected area diffraction, as represented in Figures 2(c) and 2(d). From the above described results, the σ phase can precipitate in the δ -ferrite after the third pass welding.

Table 2 showed the TEM-EDS result that the purpose is to analyze the compositions of δ -ferrite and σ phase in the third pass fusion zone (304-2). The σ phase had a higher Cr and a lower Si contents than δ -ferrite. This is because that the precipitation of σ phase in a high Cr region of δ -ferrite can consume Si content. Hence, the Cr content had a main role in forming the σ phase, and Si had an enhanced effect on the precipitation of σ phase.

3.2. Diffusion Calculation. The formation of a σ phase includes temperature and elemental diffusion. Hence, this section will determine the precipitation temperature of a σ phase using a differential scanning calorimeter (DSC) in an AISI 304 multipass welding system, as shown in Figure 3. The diagram shows two exothermic and two endothermic peaks with peak temperatures of 861°C , 934°C , 678°C , and 893°C , respectively. This is for the purpose of plotting the composition of the σ phase in this study (35 wt% Cr, 4 wt% Ni) to the Fe-Cr-Ni phase diagram shown in Figure 4. Here, the solidification order was $L \rightarrow \delta + L \rightarrow \delta \rightarrow \delta + \gamma \rightarrow \gamma + \delta + \sigma$. The above-mentioned results are marked out in Figure 4. It was determined that the σ phase precipitates from 400°C to 934°C .

3.3. Calculation of the General Diffusion Equation. In the previous published paper [19], the diffusivities D_{Cr}^{δ} , D_{Ni}^{δ} , D_{Ni}^{γ} , and D_{Cr}^{γ} were calculated at various peak temperatures (678°C , 861°C , 893°C , and 934°C), and the $\delta \rightarrow \sigma + \gamma_2$ eutectoid decomposition was discussed in accordance with the Vitek diffusion equation. In this section, the general diffusion equation calculation will be compared with the Vitek general equation. Its primary purpose is to examine the differences between the general diffusion and the Vitek diffusion equation. The general diffusion equation is shown

TABLE 3: Diffusion coefficients of elements of the δ and γ phases in stainless steel.

Element	Diffusion coefficient (cm ² /sec)									
	$\delta(\alpha)$ -iron					γ -iron				
	Temperature (°C)					Temperature (°C)				
	20	400	800	1100	1400	20	400	800	1100	1400
$\delta(\gamma)$ -Fe	10^{-46}	10^{-19}	10^{-12}	10^{-9}	10^{-7}	10^{-53}	10^{-22}	10^{-14}	10^{-11}	10^{-9}
Al			10^{-11}	10^{-9}	10^{-7}	10^{-36}	10^{-15}	10^{-9}	10^{-8}	
B						10^{-20}	10^{-9}	10^{-6}	10^{-5}	
C	10^{-17}	10^{-8}	10^{-5}	10^{-4}		10^{-27}	10^{-13}	10^{-8}	10^{-6}	
Cr	10^{-42}	10^{-18}	10^{-11}	10^{-9}	10^{-7}	10^{-37}	10^{-18}	10^{-13}	10^{-11}	10^{-9}
Co	10^{-44}	10^{-19}	10^{-12}	10^{-10}		10^{-68}	10^{-26}	10^{-16}	10^{-12}	
Cu			10^{-11}	10^{-9}		10^{-49}	10^{-20}	10^{-12}	10^{-10}	
H	10^{-5}	10^{-3}	10^{-3}	10^{-3}		10^{-10}	10^{-5}	10^{-4}	10^{-3}	
Mn						10^{-53}	10^{-22}	10^{-14}	10^{-11}	10^{-9}
Mo	10^{-46}	10^{-18}	10^{-11}	10^{-9}	10^{-7}	10^{-49}	10^{-21}	10^{-13}	10^{-11}	10^{-9}
N	10^{-17}	10^{-8}	10^{-6}	10^{-5}		10^{-31}	10^{-13}	10^{-8}	10^{-7}	
Nb								10^{-13}	10^{-11}	10^{-9}
Ni	10^{-45}	10^{-18}	10^{-11}	10^{-9}		10^{-54}	10^{-23}	10^{-15}	10^{-11}	10^{-9}
P	10^{-34}	10^{-12}	10^{-8}	10^{-7}	10^{-6}			10^{-14}	10^{-9}	10^{-8}
S	10^{-34}	10^{-12}	10^{-8}	10^{-7}	10^{-6}			10^{-14}	10^{-9}	10^{-8}
Si	10^{-50}	10^{-21}	10^{-13}	10^{-11}	10^{-7}	10^{-48}	10^{-20}	10^{-13}	10^{-12}	10^{-9}
Ti	10^{-47}	10^{-19}	10^{-12}	10^{-9}	10^{-7}	10^{-49}	10^{-21}	10^{-13}	10^{-11}	10^{-9}
V			10^{-12}	10^{-9}	10^{-7}			10^{-14}	10^{-11}	10^{-9}
W	10^{-60}	10^{-26}	10^{-17}	10^{-10}		10^{-68}	10^{-30}	10^{-20}	10^{-16}

FIGURE 4: The Fe-Cr-Ni phase diagram (60% Cr).

obtained. By calculating (9), the diffusion activation energy was calculated:

$$\ln(10^{-2}) = \ln[\exp(-2.44 \times 10^{-5}Q)], \quad (8)$$

$$-4.605 = -2.44 \times 10^{-5}Q, \quad (9)$$

then $Q = 188729 \text{ KJ} \cdot \text{mole}^{-1}$.

The $Q = 188729 \text{ KJ} \cdot \text{mole}^{-1}$ and 678°C were substituted into (3) and (1), respectively. Hence, (10) and (11) were obtained.

One equation was created from (10) and (11) and sorted simultaneously, as shown in (12)–(14). Finally, the diffusivity of Cr in δ -ferrite at 678°C ($D_{\text{Cr}(678^\circ\text{C})}^\delta$) can be calculated.

In accordance with the above calculated method, a series of diffusivities (D_{Cr}^δ , D_{Ni}^δ , D_{Ni}^γ , D_{Cr}^γ , D_{Si}^δ , and D_{Si}^γ) in the δ and γ phases at different peak temperatures (861°C , 893°C , and 934°C). The calculated results are listed in Table 4. $D_{\text{Cr}}^\delta = D_{\text{Ni}}^\delta$, indicates that it held the same diffusivity ($6.58 \times 10^{-13} \text{ cm}^2 \cdot \text{sec}^{-1}$) of Cr and Ni in δ -ferrite during the $\delta \rightarrow \sigma + \gamma_2$ eutectoid decomposition. Therefore, the Cr and Ni were easier to diffuse in the δ -ferrite than in the γ -phase. This can be attributed to higher (D_{Cr}^δ and D_{Ni}^δ) and lower diffusivities (D_{Ni}^γ). Consequently, the σ phase and secondary austenite (γ_2) were precipitated in the δ -ferrite particles in

TABLE 4: Diffusivities of the elements of Cr, Ni, and Si in δ -ferrite and γ -austenite at various peak temperatures in accordance with the general diffusion equation (D_{general}).

Temperature ($^{\circ}\text{C}$)	Diffusivity (cm^2/sec)					
	D_{Cr}^{δ}	D_{Ni}^{γ}	D_{Ni}^{δ}	D_{Cr}^{γ}	D_{Si}^{δ}	D_{Si}^{γ}
678	6.58×10^{-13}	4.38×10^{-18}	6.58×10^{-13}	6.58×10^{-15}	6.39×10^{-15}	2.56×10^{-14}
861	3.09×10^{-11}	9.26×10^{-15}	3.09×10^{-11}	3.09×10^{-13}	3.13×10^{-13}	1.75×10^{-13}
893	5.36×10^{-11}	3.33×10^{-14}	5.36×10^{-11}	5.37×10^{-13}	5.46×10^{-13}	2.32×10^{-13}
934	1.04×10^{-10}	1.08×10^{-13}	1.04×10^{-10}	1.04×10^{-12}	1.08×10^{-12}	3.23×10^{-13}

TABLE 5: Jump frequencies of the solute atoms of Cr, Ni, and Si in δ and γ at various peak temperatures.

Temperature ($^{\circ}\text{C}$)	Jump frequencies of solute atoms (sec^{-1})					
	$\Gamma_{\text{Cr}}^{\delta}$	$\Gamma_{\text{Ni}}^{\gamma}$	$\Gamma_{\text{Ni}}^{\delta}$	$\Gamma_{\text{Cr}}^{\gamma}$	$\Gamma_{\text{Si}}^{\delta}$	$\Gamma_{\text{Si}}^{\gamma}$
678	6370	0.04	6370	61	62	238
861	299000	90	299000	2877	3040	1629
893	518000	310	518000	5001	5307	2165
934	1012317	1004	1012317	9695	10400	3003

the third pass fusion zone. However, there is no prediction for the diffusivity of Si in the δ -ferrite and γ phase (D_{Si}^{δ} and D_{Si}^{γ}) in regard to the Vitek diffusion equation. The empirical formula for Si in the δ and γ phases was derived from the general diffusion equation, as illustrated in (15) and (16). Table 4 shows the $D_{\text{Si}}^{\delta} > D_{\text{Si}}^{\gamma}$ indicating that Si had a higher diffusion rate in δ -ferrite than in the γ -phase. However, the Si can promote $\delta \rightarrow \sigma + \gamma_2$ eutectoid decomposition from the above-described result. This is the primary reason that precipitation of the σ phase occurs through a Fe-Cr-Si-rich intermetallic compound in the third pass fusion zone of this study:

$$10^{-11} = D_0 \cdot \exp\left[\frac{-188729}{8.314(800 + 273)}\right], \quad (10)$$

$$D_{\text{Cr}(678^{\circ}\text{C})}^{\delta} = D_0 \cdot \exp\left[\frac{-188729}{8.314(677.9 + 273)}\right], \quad (11)$$

$$\frac{10^{-11}}{D_{\text{Cr}(678^{\circ}\text{C})}^{\delta}} = \exp(-21.15 + 23.87), \quad (12)$$

$$D_{\text{Cr}(678^{\circ}\text{C})}^{\delta} = \frac{10^{-11}}{\exp(-21.15 + 23.87)}, \quad (13)$$

$$D_{\text{Cr}(678^{\circ}\text{C})}^{\delta} = \frac{10^{-11}}{15.18}, \quad (14)$$

$$\text{then } D_{\text{Cr}(678^{\circ}\text{C})}^{\delta} = 6.58 \times 10^{-13} \text{ cm}^2 \cdot \text{sec}^{-1},$$

$$D_{\text{Si}}^{\delta} = \exp\left(-\frac{22700}{T}\right), \quad (15)$$

$$D_{\text{Si}}^{\gamma} = \exp\left(-\frac{11337}{T}\right). \quad (16)$$

3.4. The Jump Frequencies of the Cr, Ni, and Si Atoms in δ and γ Phases. Atom jump frequency (Γ) can be calculated using the above-mentioned calculation of diffusivities. This section will explain the mechanisms of eutectoid decomposition

from an atom jump perspective. Atom jump frequency can be calculated as shown in the following equation:

$$\Gamma = \frac{6D}{\lambda^2}, \quad (17)$$

where Γ is the jump frequency of solute atom (sec^{-1}), D is the diffusion coefficient ($\text{cm}^2 \cdot \text{sec}^{-1}$), and λ represents space between the two atoms (nm).

Because the atom spacing of the δ -ferrite and γ phase is $\sqrt{3}a/2$ and $\sqrt{2}a/2$, respectively, it is understood that the lattice constants of the δ -ferrite and γ phases are about 2.87 nm and 0.359 nm. Then, the values of diffusivities from Table 5 were substituted into (17), and a series of the jump frequencies were obtained, as shown in Table 4. This Table shows $\Gamma_{\text{Cr}}^{\delta} = \Gamma_{\text{Ni}}^{\delta}$ in the third pass fusion zone. This means that the solute atoms of Cr and Ni had a higher atom jump frequency with a temperature range of 678 $^{\circ}\text{C}$, 861 $^{\circ}\text{C}$, 893 $^{\circ}\text{C}$, and 934 $^{\circ}\text{C}$ in the eutectoid decomposition of $\delta \rightarrow \sigma + \gamma_2$, as shown in Figure 5. This tendency is in agreement with previous results ($D_{\text{Cr}}^{\delta} = D_{\text{Ni}}^{\delta}$). Especially notable at 934 $^{\circ}\text{C}$, the jump frequencies of Cr and Ni solute atoms were about 10^7 times per second in δ -ferrite in the third pass fusion zone. Conversely, the jump frequency of the Ni solute atom in the γ phase was very small (just 1004 times) at 934 $^{\circ}\text{C}$, as shown in Figure 6. Hence, the σ phase and secondary austenite (γ_2) formed easily in δ -ferrite. Furthermore, the jump frequency of the Si solute atom in δ -ferrite ($\Gamma_{\text{Si}}^{\delta}$) was higher than that of Cr and Ni in δ -ferrite ($\Gamma_{\text{Cr}}^{\delta}$ and $\Gamma_{\text{Ni}}^{\delta}$). As shown in Figure 7, the diffusivity of the Si solute atom increased with an increase in the jump frequency of the Si solute at various peak temperatures. Hence, the Si can be understood to accelerate the eutectoid decomposition of $\delta \rightarrow \sigma + \gamma_2$.

3.5. Modified Vitek Diffusion Equation. As previous Hsieh and Wu's study in Table 6 [19], the difference in diffusivities between the D_{Cr}^{δ} and D_{Ni}^{δ} was very small according to the Vitek model calculation. This is despite the fact that

TABLE 6: Diffusivities of the Cr and Ni in δ -ferrite and γ -austenite at various peak temperatures in accordance with the Vitek model.

Peak temperature ($^{\circ}\text{C}$)	Diffusivity ($\text{cm}^2 \cdot \text{sec}^{-1}$)			
	D_{Cr}^{δ}	D_{Ni}^{γ}	D_{Ni}^{δ}	D_{Cr}^{γ}
678	1.31×10^{-13}	1.11×10^{-16}	1.00×10^{-13}	3.78×10^{-16}
861	1.74×10^{-11}	4.73×10^{-14}	1.34×10^{-11}	1.60×10^{-13}
893	3.52×10^{-11}	1.13×10^{-13}	2.71×10^{-11}	3.84×10^{-13}
934	8.14×10^{-11}	3.18×10^{-13}	6.26×10^{-11}	1.08×10^{-12}

FIGURE 5: The diffusivities of D_{Cr}^{δ} and D_{Ni}^{δ} and the jump frequencies at various peak temperatures. FIGURE 7: The diffusivity of D_{Si}^{δ} and the jump frequencies at various peak temperatures. FIGURE 6: The diffusivity of D_{Ni}^{γ} and the jump frequencies at various peak temperatures.

the order of the D_{Cr}^{δ} is equivalent to the D_{Ni}^{δ} . Generally speaking, the general diffusion equation is more precise than other diffusion empirical equations. For this study, the Vitek diffusion equation was modified and its accuracy improved, as indicated in (18). The modifications are listed in Table 7 where it is shown that the modified coefficient had a greater at 678°C than other peak temperatures. In total, the difference between the general diffusion equation and

TABLE 7: Modified diffusion equation at various peak temperatures.

Temperature ($^{\circ}\text{C}$)	Coefficient			
	C_{Cr}^{δ}	C_{Ni}^{γ}	C_{Ni}^{δ}	C_{Cr}^{γ}
678	5.02	0.33	6.58	17.4
861	1.77	0.19	2.30	1.93
893	1.52	0.29	1.98	1.39
934	1.27	0.34	1.66	0.96

* $(D)_{\text{General}} = (C) \times (D)_{\text{Vitek}}$.

the Vitek diffusion equation did not exceed 10^2 . The Vitek model showed a quite accuracy for predicting the diffusion coefficient; however, the modified Vitek model can increase its precision:

$$(D)_{\text{General}} = (C) \times (D)_{\text{Vitek}}, \quad (18)$$

where $(D)_{\text{General}}$ is a diffusivity of general diffusion equation calculation ($\text{cm}^2 \cdot \text{sec}^{-1}$), (C) is a modified coefficient, and $(D)_{\text{Vitek}}$ is a diffusivity of Vitek diffusion model calculation ($\text{cm}^2 \cdot \text{sec}^{-1}$).

4. Conclusions

In this study, a new method for the examination of the $\delta \rightarrow \sigma + \gamma_2$ phase transformation within multipass fusion zones was investigated. The new method employed the general

diffusion equation with comparison to the Vitek diffusion model during multipass dissimilar stainless steel welding. The significant results were as follows.

- (1) The precipitation of the σ phase in the δ -ferrite was observed using TEM-SAD and TEM-EDS after the multipass welding.
- (2) The diffusion coefficients of Si in δ -ferrite and γ -austenite were predicted using the general diffusion equation with comparison to the Vitek diffusion model.
- (3) The Vitek diffusion equation was modified as in the following equation: $(D)_{\text{General}} = (C) \times (D)_{\text{Vitek}}$ in the multipass fusion zone.

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