Corrosion Behaviour of High-Chromium White Iron Hardfacing Alloys in an Alkaline Solution

Varmaa Marimuthu¹ · Krishnan Kannoorpatti¹

Received: 11 July 2016/Revised: 11 September 2016/Accepted: 19 September 2016/Published online: 23 September 2016
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Abstract Hardfacing alloys of high-chromium white irons (HCWI) deposited using welding are well known for their wear resistance. These materials are applied in conditions needing not only wear resistance but also corrosion. In this study, the HCWI alloys were exposed to alkaline solutions of pH 14. Anodic polarization technique was used to study the corrosion behaviour. The experimental results found that corrosion occurs on carbides in preference to the matrix. Pourbaix diagrams of carbides were found to be very useful in explaining the corrosion behaviour of the hardfacing alloys. The significance of carbide corrosion to the use of hardfacing alloys is discussed. Possible methods to mitigate corrosion are proposed.

Keywords Hardfacing · High-chromium white irons · Alkaline · Alumina refineries · Pourbaix diagram of carbides

1 Introduction

High-chromium white irons (HCWI) are commonly used for the slurry pump components and pipelines in the Bayer refining process of alumina refineries [1, 2] due to their high wear resistance. The Bayer process solution is highly alkaline, and there are limited studies on the corrosion behaviour of the hardfacing alloys. There are also limited studies in the use of Pourbaix diagrams to predict a trend for the corrosion behaviour of hardfacing alloys.

Nelson [2] exposed hardfacing alloys consisting of Cr_7C_3 primary and eutectic carbides, and eutectic austenite to sodium hydroxide (pH 14) at 90 °C and found that the eutectic austenite corroded. Cast white irons were found to have better corrosion resistance than welds. Similarly, other investigators [1, 3] also found that the corrosion of white chromium cast irons occurred in the eutectic austenite or martensite. In contrast to this, in the study conducted by Salasi [4], on cast primary chromium carbides were found to corrode in preference to the eutectic austenite or martensite matrix. Tsai et al. [5] performed corrosion experiments on sintered pure chromium carbides (Cr_{23}C_6) 0.1 N NaOH that was deaerated and saturated with H_2 before each experiment in the temperatures range of 275–325 °C. It was found that dissolution of chromium carbides occurred.

Salasi et al. [6, 7] found that in an alkaline solution of pH 12, a layer of Fe_3O_4 can form on carbides and a passive layer forms in the matrix eutectic austenite. In the studies conducted by Nelson [2] and Mcleod [8], in alkaline solution, it was proposed that the dissolution of the matrix occurred primarily due to depletion of chromium in a matrix. They also proposed that chromium depletion hindered the formation of passive film on the surface of austenite. The corrosion of either matrix [2, 8] or carbides [6, 7] depended on the formation of passive film on the matrix. Aziz et al. [3] proposed that the corrosion attack of the matrix in an alkaline solution occurred due to micro-galvanic interaction between matrix and the carbides, and chromium depletion. Similar ideas were also proposed by other investigators [2, 6, 8, 9].

Whilst these studies have done much to improve our knowledge on corrosion behaviour of high-chromium white irons, they did not discuss a method to predict corrosion behaviour. This is where the use of Pourbaix

✉ Varmaa Marimuthu
varmaa.marimuthu@cdu.edu.au

¹ School of Engineering and IT, Charles Darwin University, Darwin, Australia

diagrams fits in. Pourbaix diagrams can not only be used to predict the corrosion behaviour of materials so that problems in the future can be avoided, but also provide simplified explanation of complicated chemistry and thermodynamics. Pourbaix diagrams can also be used to diagnose past and present problems in a thoughtful manner. They can also be used to design suitable experiments in an electrolyte of interest. Chen et al. [10] have used the Pourbaix diagrams of metals to explain the behaviour of corrosion of metals at elevated temperatures in energy industries. With this in view, Varmaa et al. [11, 12] developed Pourbaix diagrams for carbides in order to explain the trend observed in the corrosion behaviour of carbides. In this experimental study, the corrosion of hardfacing alloys based on HCWI will be explained through the use of Pourbaix diagrams of carbides.

2 Experimental Method

Hardfacing alloys based on HCWI were deposited on low carbon steel using shielded metal arc welding (SMAW). The typical composition of the deposited hardfacing alloys is given in Table 1. Alloy 1 mainly had high carbon and chromium while Alloy 2 had a number of elements, such as niobium, molybdenum and tungsten. These elements would form carbides in preference to chromium, and hence this sample was likely to contain complex carbides. The recommended welding parameters provided by the manufacturer of the hardfacing alloys are given in Table 2. To avoid the dilution, welding was performed in four layers. Samples were cut from the weld deposits and were prepared for microstructural observations. The samples were etched in 20 ml distilled water, 10 ml HCl, 30 ml HNO₃ and 5 g of FeCl₃ [13].

Corrosion tests were conducted using electrochemical methods with a Gamry Reference 3000 potentiostat and a three-electrode cell. The HCWI (sample area = 2.85 cm²) was the working electrode exposed to the electrolyte 2 M NaOH solution of pH $\approx$ 14. The graphite and silver/silver chloride were the counter and reference electrodes, respectively. Before measuring corrosion potential, the alkaline solution in the corrosion cell was deaerated using nitrogen gas for 30 min, as described in the ASTM Standard G5-94 [14]. The open-circuit potential (E_{corr}) was measured after 10 min, and then the potentiodynamic test

was conducted at the scan rate of 0.17 mV/s [14]. The potentiodynamic test was continued till the sample showed transpassive behaviour. Then, the microstructure was examined using an optical microscope by removing the corrosion product using rubber cork under running tap water [2]. The potentiodynamic tests were conducted three times for each specimen to determine the consistency of the curves.

On the basis of the potentiodynamic test results, potentiostatic etching was conducted at different potentials on HCWI samples to determine the effect of corrosion in the microstructures. The potentials, points 1–6, were selected based on the changes in the anodic polarization curves or approximately in the middle of an anodic curve segment if there was no change in the anodic polarization curves. The intention was to cover the anodic polarization curve reasonably. The points 1–6 are given in Table 3, and the microstructures of the samples were recorded. Tafel plots were also conducted at -150 – $+150$ mV with the scan rate of 0.2 mV/s to determine the corrosion behaviour. The electrochemical data analysis was done using Echem Analyst software [15].

The scanning electron microscopy (SEM, Philips XL 30 series) was used to study the microstructure, and energy dispersive X-ray spectrometry (EDS) was used study the chemical composition of the samples before and after corrosion. The ICPMS was conducted to analyse the chemical composition of the electrolyte after the corrosion tests.

3 Results

3.1 Microstructure Characteristics of HCWI Alloys

Figure 1 shows the microstructure of alloy 1 hardfacing sample. The microstructure shows primary carbides surrounded by eutectic carbides and austenite [16, 17]. The primary carbides are polygonal and some are needle shaped depending on the direction of solidification. The primary and eutectic carbides are of the type M_7C_3 and $M_{23}C_6$, respectively [18–24]. Figure 2 shows the microstructure of alloy 2 sample. The microstructure shows again primary carbides surrounded by eutectic carbides and austenite. The microstructure also shows some light regions of niobium carbide (NbC) [21].

The SEM pictures of the alloy 1 and alloy 2 samples are given in Fig. 3i, ii. The point elemental analysis was done on primary carbides (a), carbide/matrix interfaces (b), matrix (c) and eutectic carbides (d), and the values are given in Table 4. In Fig. 3i, ii, the areas marked d1 were found to be molybdenum carbide. In Fig. 3ii, the area marked a1 was found to be niobium carbide. From Table 4,

Table 1 Chemical compositions (wt%) of hardfacing alloys

Alloy type	C	Cr	Mn	V	Si	Ni	Mo	W	Nb	Fe
Alloy 1	4.8	30.0	1.1	0.2	1.4	0.5	1.7	—	—	Bal.
Alloy 2	4.2	22.6	0.2	1.7	2.0	—	5.8	2.2	5.0	Bal.

Table 2 Welding parameters

Welding type	Welding voltage (V)	Welding current (A)	Welding speed (mm/min)	Depositing numbers
MMAW	32 V	100–150 A	120–200 mm/min	4 Layers, 5 beads

Table 3 Potentials used for potentiostatic tests from the anodic curves of alloy 1 and 2

Sample	1	2	3	4	5	6
Alloy 1	−0.200	0	0.100	0.200	0.400	0.600
Potential (V) versus Ag/AgCl						
Alloy 2	−0.200	0.100	0.200	0.400	0.600	–
Potential (V) versus Ag/AgCl						

Fig. 1 Microstructure of alloy 1 sample showing polygonal primary carbides surrounded by eutectic carbides and austenite **Fig. 2** Microstructure of alloy 2 sample showing polygonal primary carbides surrounded by eutectic carbides and austenite. a1 is NbC

Fig. 3 SEM microstructure analysis of **i** alloy 1 and **ii** alloy 2 hardfacing samples

Table 4 EDS analysis (wt%) for hardfacing samples

Samples	Cr	Fe	Nb	Mo
Alloy 1				
a	46.49	37	–	–
b	10	69.74	–	–
c	7.4	80.08	–	–
d	32.57	42.04	–	–
d1	11.7	26.74	–	32.91
Alloy 2				
a	49.93	24.55	–	–
a1	–	–	65.75	–
b	14.38	68.95	–	–
c	13.07	79.48	–	–
d	53.99	33.96	–	–
d1	20.52	56.33	–	17.39

a primary carbides, b carbide/matrix interface, c matrix and d, d1 eutectic carbides

it can be seen that the primary and eutectic carbides (a and d, respectively) had high amounts of chromium and iron in both the samples. Area b denotes carbide–austenite interface and shows that the chromium is lower than 10 % for alloy 1 sample and 14.38 % for alloy 2 sample. Area c denotes austenite for both alloy 1 and alloy 2 samples, where the percentage of chromium is higher in alloy 2 samples than in alloy 1. This may be due to the presence of strong carbide-forming element, niobium, that allows chromium to remain in austenite [25]. The area d1 of both alloy 1 and alloy 2 samples exhibits the presence of molybdenum in eutectic carbide with a lower amount of chromium compared to area d.

3.2 Corrosion Behaviour of HCWI Alloys

3.2.1 Potentiodynamic and Potentiostatic Test

The potentiodynamic tests were conducted by exposing the HCWI samples in 2 M NaOH solution of pH 14. The results of the three potentiodynamic tests are given Figs. 4 and 5 for alloy 1 and alloy 2 samples, respectively. It can be seen that there was very little difference between the three tests for both the samples. The curves did not exhibit a typical active–passive behaviour but exhibited an active behaviour all through. Although the curves were very nearly the same for both alloy 1 and alloy 2 samples, there were a few minor differences. The transpassive region occurred at about 550 mV for alloy 1 samples, while it occurred at about 600 mV for the alloy 2 samples.

The microstructures after the potentiostatic etching are shown in Figs. 4 and 5. For the alloy 1 sample, corrosion occurs at the eutectic carbides at points 1–4 in Fig. 4 followed by the corrosion of primary carbides at points 5 and 6, as shown in the microstructures of Fig. 2. The microstructure of eutectic carbide corrosion is shown in Fig. 6, and one can see that the primary carbides are still intact. Figure 7 shows the microstructure of the alloy 1 sample at point 6 of the polarization curve in Fig. 4. It can be seen that the carbides have dissolved leaving just the eutectic austenite without corrosion.

For alloy 2 samples at point 3 in Fig. 5, niobium carbides have undergone extensive corrosion, as seen in Fig. 8. At the transpassive region in Fig. 5, it can be seen that the primary and eutectic carbides have undergone corrosion, as seen in Fig. 9. In summary, it can be said that primary carbide dissolution happens at higher oxidation potentials than the eutectic carbides and niobium carbides.

Fig. 4 Anodic polarization curve for alloy 1

The dissolution of primary carbides also occurred only at potentials 5 and 6 in Figs. 4 and 5. Even at these high potentials, eutectic austenite is protected. The final potentials of the work from Nelson [2] and Salasi [6] are given in Table 5. These potentials are then placed on the potentiodynamic curves in Fig. 10. Nelson [2] found that corrosion occurred mainly on eutectic austenite. The final potential of transpassive region for Nelson [2] which was placed in Fig. 10 shows that it was closer to potentiostatic test point 4 where eutectic carbides were found to corrode. However, it must be noted that Nelson did his corrosion tests at 90 °C. Although it is expected that the Pourbaix diagram regions will shift to lower potentials at high temperatures, it is interesting to note that tests conducted by Nelson [2] did not reach transpassive potential found in the present

study. This is possibly the reason why corrosion did not occur in the carbides. However, Tsai et al. [5] found that chromium carbide (Cr_{23}C_6) did corrode at 300 °C. Cr_{23}C_6 is a less stable carbide than Cr_7C_3 [11]. The final potential from the tests done by Salasi [6] was closely matching with that of alloy 1 in this investigation. The microstructures from the work of Salasi [6] also showed that corrosion occurred on eutectic and primary carbides at the transpassive regions.

3.2.2 Tafel Plot

The Tafel analysis was done on Echem Analyst to obtain the values of the slope of tangents (β_A and β_C), E_{corr} and

Fig. 5 Anodic polarization curve for alloy 2

I_{corr} . From the derived values of β_A , β_C and I_{corr} for all the HCWI samples, the polarization resistance R_p values were determined from the below equation [26]

$$\text{Polarization resistance } (R_p) = \frac{\beta_A \beta_C}{2.3 I_{\text{corr}} (\beta_A + \beta_C)}.$$

The values, E_{corr} , I_{corr} , R_p and corrosion rate, are given in Table 6. The alloy 2 specimen shows better corrosion resistance than alloy 1, due to its higher corrosion potential and polarization resistance and lower corrosion current. The better corrosion resistance of alloy 2 sample may be due to the higher percentage of chromium in matrix than alloy 1 sample as shown in Table 4a.

3.2.3 Microstructure Analysis After Corrosion

The SEM/EDS analysis was done after the corrosion test for alloy 1 sample to understand the dissolution/corrosion of elements associated in the corrosion of carbides as shown in Fig. 11. In Tables 4 and 7, the analysis of primary and eutectic carbides of alloy 1 sample before and after corrosion shows that significant dissolution of chromium element takes place at higher potentials in alkaline solution. The ICPMS tested results for alloy 1 sample in Table 8 revealed that significant dissolution of chromium (7600 $\mu\text{g/L}$) takes place at higher potentials in 2 M NaOH solution. There were no significant differences in

Fig. 6 Corrosion of eutectic carbides in alloy 1 sample

Fig. 7 Corrosion of eutectic and primary carbides in alloy 1 sample and SEM/EDS microstructure analysis of matrix sample (a) higher magnification of dissolved carbides

percentage of chromium in the matrix after the corrosion test as given in Table 7c. The increase in percentage of iron in the carbides after potentiostatic corrosion test is clearly seen in Table 7 of alloy 1 sample, and this may be due to

passive film growth and low dissolution of iron in alkaline solutions [27]. The partial dissolution of iron 3330 $\mu\text{g/L}$ was detected using ICPMS. The EDS and ICPMS test results also showed that there was complete dissolution of

Fig. 8 Corrosion of niobium carbide in alloy 2 sample

Fig. 9 Corrosion of NbC, eutectic and primary carbides in alloy 2 sample

Table 5 The final potentials used for potentiodynamic test of HCWI at alkaline solutions conducted by other literature

Other literature	Final potentials versus Ag/AgCl
Nelson [2]	0.22
Salasi [6]	0.600

molybdenum, manganese and vanadium elements at higher potentials in 2 M NaOH solution as given in Tables 7 and 8. After corrosion, the carbides become hollow in shape, and some cracks were found as shown in Fig. 7a. Hardness test was done on the alloy 1 sample before and after

corrosion as shown in Fig. 12 to ascertain how corrosion affected carbides. This will help us understand how useful is the carbide to resist wear. It was found that there was a significant drop of hardness from 2299 HV before corrosion to 41.8 HV after corrosion in the carbides. This clearly showed that the carbides had indeed corroded at higher potentials in an alkaline solution and perhaps are not useful any longer to resist wear.

3.2.4 Corrosion Analysis of Carbide and Matrix Samples

After the carbides had dissolved in the potentiostatic tests, the potential of the eutectic austenite alone in the alkaline solution was measured for alloy 1 sample. For this, epoxy

Fig. 10 Final potentials (FP) used for potentiodynamic test of HCWI at alkaline solutions conducted by other literature

Table 6 I_{corr} , E_{corr} and corrosion rate obtained from Tafel plot for hardfacing samples

Experimental determinations	Alloy 1	Alloy 2
I_{corr} (μA)	4.68	1.61
E_{corr} (V) versus Ag/AgCl	-0.522	-0.427
E_{corr} (V) versus SHE	-0.3	-0.205
R_p ($\Omega \text{ cm}^2$)	5.92×10^{-3}	0.0264
Corrosion rate (mm/year)	0.0117	0.0042

solution was placed on the surface of the sample, and the sample was repolished after the epoxy hardened. Similarly, in order to measure the potential of the carbides, the matrix was dissolved in an acidic solution [28]. Again, epoxy was

applied to the sample surface, and it was repolished. After preparing carbide and matrix sample, corrosion potential of the samples was recorded in 2 M NaOH alkaline solution as shown in Table 9. The matrix sample shows nobler potential than a carbide sample in highly alkaline solution.

4 Discussion

4.1 Galvanic Effect

The results showed that corrosion of HCWI in alkaline solution occurs mainly on carbides. The corrosion of the HCWI can be explained by micro-galvanic corrosion between carbide and eutectic austenite [3]. It was shown in this work that carbide exhibited a less noble potential than

Fig. 11 SEM microstructure analysis of alloy 1 hardfacing sample after corrosion

Table 7 EDS analysis (wt%) of alloy 1 sample after corrosion

Sample	Cr	Fe
Alloy 1		
a	2.08	48.21
b	10.71	80.03
c	10.89	79.16
d	7.72	70.42
a primary carbides, b carbide/matrix interface, c matrix and d eutectic carbides		

Table 8 ICPMS Analysis in 2 M NaOH solution after corrosion test at point 6 of alloy 1 sample

Cr (μg/L)	Fe (μg/L)	Mo (μg/L)	Mn (μg/L)	V (μg/L)
7600	3330	324	201	75.5

the eutectic austenite. Galvanic couple is formed between carbides and matrix. The noble potential of eutectic austenite matrix produces a cathode, and active potential of eutectic and primary carbides produces an anode. Due to potential difference, preferential dissolution of carbides occurs in preference to the matrix. Table 7 also confirms for alloy 1 sample, after corrosion test, that there is significant reduction of chromium content in the carbides but not in the matrix. Others [2, 6, 8, 9] have found similar galvanic corrosion effect for HCWI materials in alkaline media. The study conducted by Tsai et al. [5] suggests that the dissolution rates of carbides are higher in alkaline solution while in contact with platinum. This theory is in line with the findings of Salasi et al. [4]. However, they contradict the findings of others who found that carbides

did not corrode [2, 3, 8]. It is possible that these investigators did not test till the transpassive region.

The corrosion trend of HCWI hardfacing deposits can be explained through the use of Pourbaix diagrams. Since the alloys have significant amounts of elements other than chromium, the superimposition of Pourbaix diagrams of other elements on chromium carbide Pourbaix diagrams will be useful in explaining the stability of these alloys in high pH solutions.

4.2 Stability of Carbides of HCWI and Pourbaix Diagrams

From the Pourbaix diagram of chromium carbide (Cr_7C_3) in Fig. 13, it can be seen at pH of 14, the immune region boundary is around -1.3 V (SHE). At higher potentials, Cr_7C_3 forms Cr_2O_3 followed by CrO_4^{2-} . The rest potentials measured for the hardfacing alloys lie in the region of Cr_2O_3 . The presence of iron (Fe) was identified in carbides and eutectic austenite as described in Table 4. There is a sort of galvanic effect described in the previous section due to the presence of Fe in carbides and eutectic austenite. It would be useful to superimpose the Fe Pourbaix diagram [29] with Pourbaix diagram of Cr_7C_3 as shown in Fig. 14.

At the rest potential of the carbides, the hardfacing alloys undergo corrosion as seen in Figs. 4 and 5. The Tafel analysis also indicates that corrosion can be identified at rest potentials for HCWI hardfacing alloys, and this is borne out by the fact that the anodic polarization curve exhibited active behaviour. The alloys undergo some mild corrosion until point 4 in Figs. 4 and 5. Although the alloys are in the region of corrosion as shown in Fig. 14, due to the presence of Fe and its oxide Fe_2O_3 , the alloys undergo only mild corrosion. The presence of Fe_2O_3 stops extensive corrosion from happening. However, on reaching the potential 5, there is substantial corrosion of carbides as Fe is also taken to the region of corrosion FeO_4^{2-} . Corrosion of carbides can be seen in the microstructures in Figs. 7 and 9.

However, in the case of alloy 2, NbC was found to have no Fe as seen in Table 4. This is the reason NbC was found to corrode at lower potentials than Cr_7C_3 primary carbides as seen in Fig. 5. On superimposing NbC and Cr_7C_3 Pourbaix diagrams on each other in Fig. 15, it can be seen that NbO is formed at the rest potentials. NbC has undergone extensive corrosion at potentiostatic point 3, whereas Cr_7C_3 was still reasonably stable until potentiostatic point 6. NbC is found to be unstable only due to the absence of iron. Strehblow et al. [30, 31], McLeod et al. [32] and Salasi [7] have found that iron forms a thicker stable passive layer than chromium in highly alkaline solution. Further at potentiostatic point 4 in the Pourbaix diagram of Fe, FeO_4^{2-} is the stable phase.

Fig. 12 Hardness test **a** before corrosion, **b** after corrosion

Table 9 Corrosion potential for carbide and matrix alloy 1 sample in 2 M NaOH solution

Sample (alloy 1)	Corrosion potential (V) versus Ag/AgCl
Carbide	−0.625
Matrix	−0.379

The molybdenum, manganese and vanadium are not stable and exist in the corrosion region at higher potentials as explained in the Pourbaix diagram of molybdenum [33], manganese [34] and vanadium [35] at pH 14. According to these Pourbaix diagrams, the elements molybdenum, manganese and vanadium may dissolve in the form of species MoO_4^{2-} , MnO_4^- and VO_4^{3-} . The ICPMS test

Fig. 13 Pourbaix diagram of in pH 14 with anodic potentials of HCWI [11]

Fig. 14 Pourbaix diagram of Cr_7C_3 -Fe in pH 14 with anodic potentials of HCWI [11, 29]

Fig. 15 Pourbaix diagram of Cr_7C_3 -NbC in pH 14 with anodic potentials of HCWI [11, 12]

results as shown in Table 7 also revealed that the dissolution of above elements occurred at higher potentials as these are located in the corrosion region.

The dissolution of carbides in the hardfacing alloy shown in this work will have an effect on the usefulness of hardfacing material against wear. The hardfacing alloys

even at open-circuit potential are in the active state in high pH solutions. Although it has been shown through Pourbaix diagrams [11] that there is the formation of Cr_2O_3 passive layer, it was argued that in order for the passive layer to form, Cr_7C_3 should undergo dissociation. It was found that iron in the primary carbides provides some stability to the carbides. However, under wear conditions, the passive layers would be removed and will likely be subjected to synergistic erosion–corrosion mechanism. Although no use of cathodic protection has been done on hardfacing alloys, it may be useful to apply cathodic potentials to place the hardfacing alloys in the immune region of the Pourbaix diagram. In that instance, the loss of material would be likely erosion and not the synergistic erosion–corrosion. Work is underway in this laboratory to introduce nitrogen into the hardfacing alloys to form nitrides, carbo-nitrides or simply nitrogen as an alloying element. Nitrogen has been added to stainless steels to improve corrosion resistance [36, 37].

By developing the Pourbaix diagrams for various carbides [12], it can be seen that the formation of passive layers will result in the dissociation of carbides, and erosive particles will likely remove the oxide layer resulting in erosion–corrosion. The dissociation of carbides to form the passive layer will likely render the carbides ineffective against wear. It was found that silicon and tungsten carbides also undergo corrosion [38, 39]. All carbides seem to have this issue. It is proposed that cathodic potentials may be another avenue that needs to be explored to improve the erosion–corrosion resistance of hardfacing alloys.

5 Conclusions

The corrosion behaviour of typical HCWI alloys with different chemical compositions in alkaline solutions was investigated. The four main conclusions drawn from this study are as follows:

- (1) The preferential corrosion occurs on carbides for all the HCWI samples at higher potentials.
- (2) Eutectic austenite attains noble potential and acts as a cathode, and the carbides attain an active potential.
- (3) The analysis of SEM/EDS and Pourbaix Diagram reveals that chromium, molybdenum and carbon are not so stable as iron at higher potentials in alkaline solutions.
- (4) Application of cathodic potentials to place the carbides in the immune region is likely to improve the durability of carbides in erosive–corrosive fluids.

Compliance with Ethical Standards

Conflicts of Interest On behalf of all authors, Varmaa Marimuthu states that there is no conflict of interest.

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