Study of helium ion-irradiated Inconal 625 alloy treated in various environments

S. Sharma[a], D. Arora[b], V. Baranwal[c], V.Yadav[d], S. K. Gupta[d], J.P. Bhamu[e], A. C.Pandey[c,f], Y. C. Sharma[g,*]

[a]*Department of Mechanical Engineering, Vivekananda Institute of Technology (East), Jaipur-303012*

[b]*Department of Mechanical Engineering, Vivekananda Institute of Technology, Jaipur-303012*

[c]*Nanotechnology Application Centre, University of Allahabad, Allahabad-211001*

[d]*Department of Physics, BanasthaliVidyapeeth, Banasthali-304002*

[e]*Department of Mechanical Engineering. Government Engineering College Bikaner, Bikaner-334004*

[f]*Inter University Accelerator Consortium (IUAC), New Delhi-110067*

[g]*Innovation, Research and Development, Dr CBS Cyber Security LLP, Jaipur-302006*

The objective of this work is to study the surface morphology and hardness of Inconal-625 alloy when the samples were exposed to different acids and bases and then these samples were irradiated with Helium ions (He^{2+}). Nickel based Inconal-625 is a super alloy which is highly used in oil industry, refineries, boilers, acid and base storage tanks, nuclear power reactors and aerospace equipments like jets and rockets,in which safety and operations are the most important considerations.In these applications the surfaces are exposed to various acids and bases and also got interacted with various ions specially helium ions. So to evaluate the interactions the samples were exposed with six different solutions i.e. H_2SO_4, KOH, NaOH, HNO_3, HCl, and Chromic Acid for time duration of 240 hrs. After that samples were irradiated with He^{2+} ions beam having the fluence of 10^{15}ions/cm^2.Microstructural modifications in the surface have been studied using SEM-EDAX (Scanning electron Microscope-Energy Dispersive X-Ray Analysis).It has been observed that amount of Niobium (Nb), Nickel (Ni), Chromium (Cr) and other major constituent elements vary in context to different environments. The Hardness of the samples has been studied using Vickers microhardness test. The hardness of the samples varies in various environments and it has been observed that value of microhardness decreases in every case as comparison to untreated sample. The maximum of the hardness has been observed in case when the sample was corroded with Chromic Acid.

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1. Introduction

Nickel-chromium based alloys especially Inconel-625 which is having excellent creep and mechanical properties at high temperature and pressure and exhibits high corrosion resistance isemployed widely in many industries like chemical, refinery, aerospace like rockets and satellites and nuclear reactors [1]. But safety is the most important concern in these sectors because handling of these alloys are quite difficult in different environments,especially in nuclear reactors and human containing vessels like international space station (ISS) of NASA, US.Although MSR (Molten Salt reactors) technology is being extensively used in nuclear reactors due to its safety features [2],but it has been observed that at higher temperatures various salts can be highly corrosive towards the structural material [9]. During the nuclear reactions it has also beenverified that irradiation of ions creates abundant defects and it leads to micro structural defects in the Nickel- chromium based super alloys [10-12].It has been observed that in nickel C-276 alloy there is a strong relation between the density of dislocation structure and irradiation doses [13].As a

[*]Corresponding author: yc.sharma.vit@gmail.com

result of irradiation of ion species, defects such as embrittlement, hardening and swelling has been observed in irradiated Ni-based alloys [14-16].It has been observed by Zhu et al. that helium ion irradiation induced bubbles and cavities in the samples which resulted in enhanced intergranular corrosion of Ni-based alloy in fluoride molten saltenvironment[17].It has been concluded that the impact of irradiation caused the structural damage which might added to corrosion [18-20]. It has been reported extensively in the nuclear engineering literature that the formation and growth of helium bubbles in irradiation microstructures [18-23] weakens the corrosion resistant nature of the materials. There are only a few reports about molten salt environments having the irradiation of the helium and helium ions and their impact on corrosion of structural material.As it was studied earlier that Nickel based alloys have the greatest resistance towards corrosion but degradation due to helium ions and helium bubbles is a still an issue [2-9]. Hence in this study the influence of helium ions on the surface morphology and hardness of Inconel-625 has been studied. Firstly samples were exposed to different corrosive environment such as H_2SO_4, KOH, NaOH, HNO_3, HCl, and Chromic Acid for the duration of 240 hrs and then helium ions irradiation wasperformed with the fluence of 10^{15}ions/cm^2.Microstrctural changes were examined by SEM (Scanning Electron Microscope) and EDAX (Energy Dispersive X-Ray Analysis). Hardness of the treated samples was examined by Vickers hardness tester.

2. Material and Methodology

2.1. Sample Preparation

Six square plates having dimensions 2.54 cm * 2.54 cm and thickness 2.54 cm were prepared. The material used in this study was provided by “Vilares Materials”. Samples were washed with distilled water and then cleaned with acetone. Glass hooks were used in corrosion experiments to hang the samples in the beakers filled with different solutions. The chemical composition of the as received super alloy is given Table 1.

Table 1.Composition of Inconel-625 alloy.

Elements, wt%						
	Ni	Cr	Mo	Nb	Fe	Ti
Inconel-625	65.8%	20.75%	8.27%	3.38%	1.04%	0.216%

3. Experimental Characterization

3.1.Corrosion of the samples

For corrosion test six different solutions were taken i.e. H_2SO_4, KOH, NaOH, HNO_3, HCl, and chromic Acid. These tests were performed at advanced materials Laboratory, VGU. Six different solutions were prepared in chemistry lab. The normality of all the solutions waskept at 1N. Samples were named as 1 to 6 as according to their exposure with different solutions as shown in Table 2.

Table 2. Sample 1 to 6 with different corrosive environments.

Sample No.	Solution	Time
1	H_2SO_4	240 hrs
2	KOH	240 hrs
3	NaOH	240 hrs
4	HNO3	240 hrs
5	HCl	240 hrs
6	Chromic Acid	240 hrs

Time taken for the exposure of samples with different solutions was 240 hrs. Samples were hanged in the beakers filled with different solutions with the help of glass hooks. A small hole was created in the corner of each sample so that glass hooks can easily support the samples to hang down.

3.2. Helium-Ion Irradiation

Since after the completion of corrosion experiments the samples were irradiated with He^{2+} ion beam having the fluence of 10^{15}ions/cm^2. The current taken was between the range 12 amp to 15 amp. With the energy 650 KeVprojected range is 1.16 micron± 144 nanometer. Electronic energy loss 70.8 eV/ angstrom (Å) and nuclear energy loss 0.164 eV/ angstrom. So it is inferred that the dominating energy loss is electronic energy loss. The samples were attached with probe and then were irradiated with He^{2+} ions. The samples attached in probe are shown in Fig.1.

Fig. 1.Sample handling & placement on Probe Bracket for Ion irradiation.

3.3. SEM-EDAX Characterization

The Surface morphology of the samples was examined by a field emission scanning electron microscope (MIRA 3 TESCAN) and the size distribution of the particles was analyzed by image J software. The variation in the chemical composition was examined by EDAX using Aztec software.

3.4. Hardness Test

The Vickers hardness tests of the samples were experimented by Vickers micro hardness tester model NEXUS 4303. A load of 500 g was applied for a dwell time of 10 secs on the surface. Three indentations were made in each sample to find the homogeneity.

4. Results and discussions

4.1. Surface morphology

The Surface morphology of the corroded and irradiated sampleswas investigated using SEM (Scanning Electron Microscope). It is quite easily seen that high grain boundary grooves with curved shape are there on the surface of all the samples as shown in Fig. 2. The depth of the grooves increases as it approaches to the grain boundaries. This leads to the result that corrosion was intergranular for all the samples.The SEM images of the samples are given in Fig. 2.

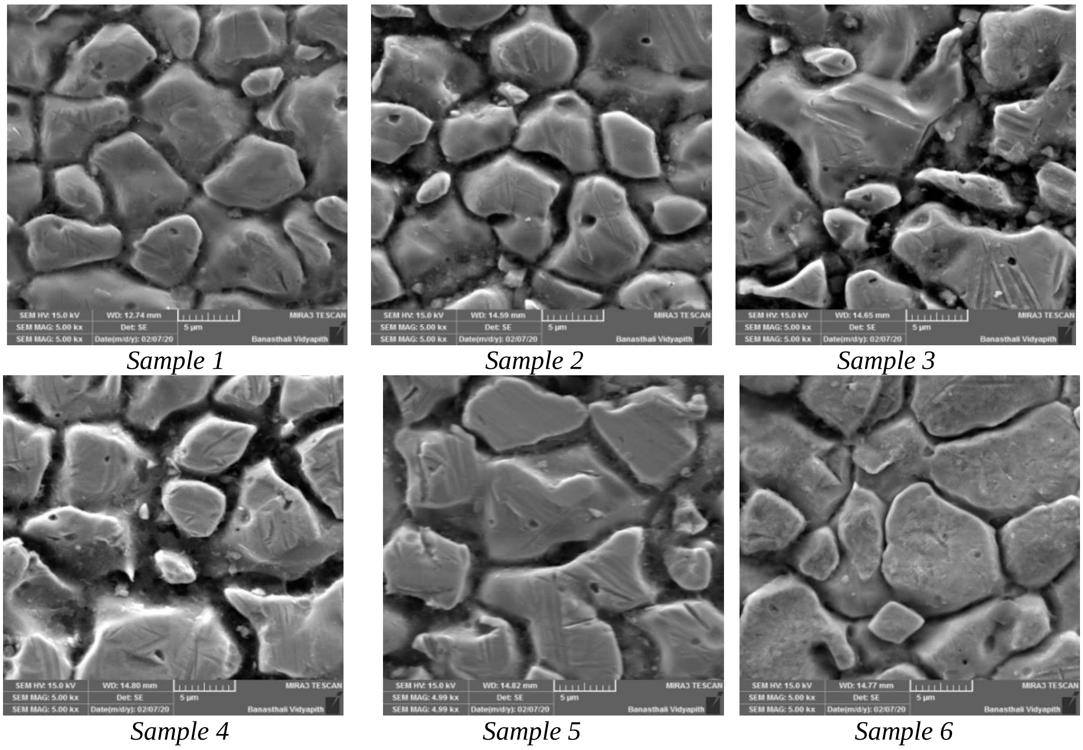
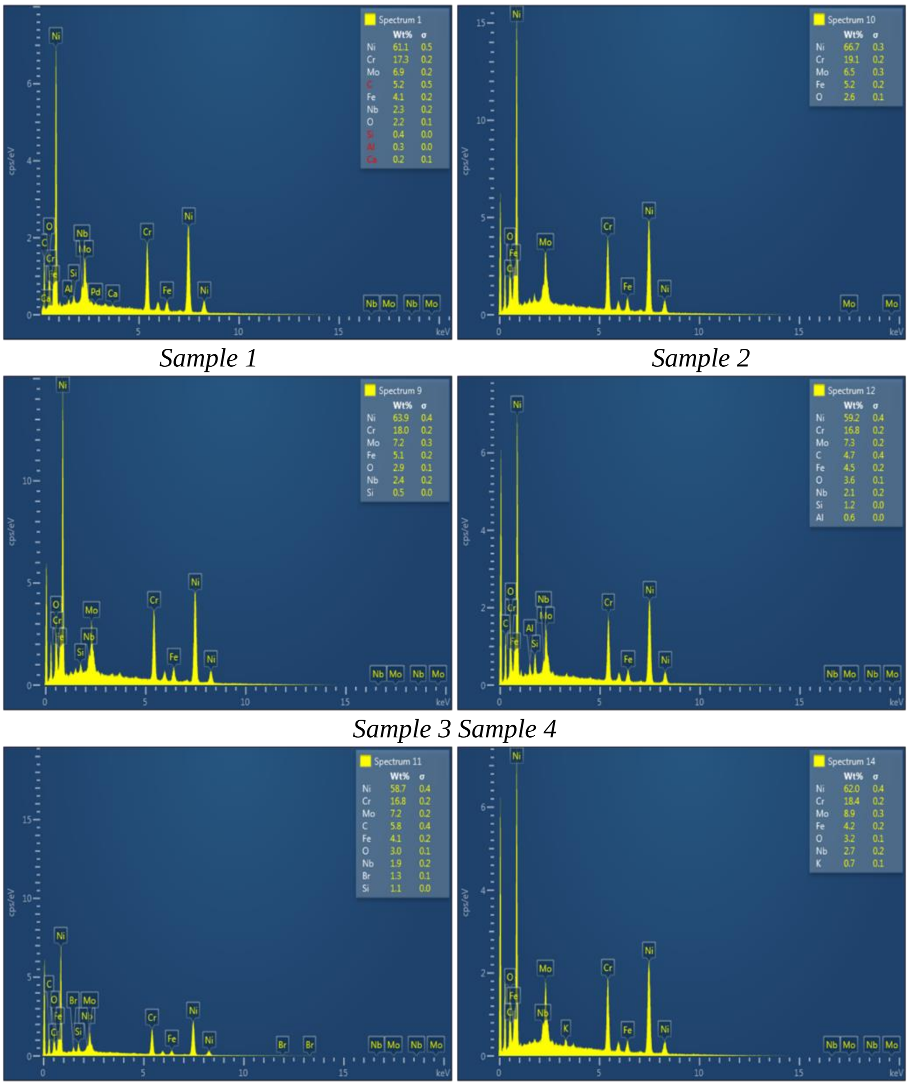


Fig. 2. SEM Photos of the treated samples.

For all the samples the surface becomes rough and cavities were observed.These results indicated that grain matrices were attacked by the various environments in all the cases and hence the helium ions irradiation enhances intragranularcorrosion. But in case of sample 5 and sample 6 which were exposed to HCl and chromic acid respectively the impact was less hence small number of pores and cavities were observed. Severe impact has been observed in case of sample of 2 and 3 which were exposed to KOH and NaOH respectively and large numbers of cavities were observed on the surface.

To identify the elemental composition of the treated samples, they were examined using EDAX

(Energy dispersive X-ray Analysis). The EDAX images of the all the samples are shown in Fig.3.



Sample 1 Sample 2

Sample 3 Sample 4

Sample 5 Sample 6

Fig. 3. EDAX Images of the samples.

It is clearly shown in the EDAX images that nominal chemical composition of the samples was changed when treated in different environment. As shown in Table 1 inconel-625 contains Nickel (Ni) and Chromium (Cr) as primary elements. Table 3 shows the fractional presence of major elements from sample 1 to 6.

Table 3. Elemental composition in % of treated samples.

Sample	% of elements				
	Ni	Cr	Mo	Nb	Fe
Untreated sample	65.8	20.75	8.27	3.38	1.04
Sample-1	61.1	17.3	6.9	2.3	4.1
Sample-2	66.7	19.1	6.5	-----	5.2
Sample-3	63.8	18.0	7.2	2.4	5.1
Sample-4	59.2	16.8	7.3	2.1	4.5
Sample-5	58.7	16.8	7.2	1.9	4.1
Sample-6	62.0	18.4	8.9	2.7	4.2

It has been observed that the increased ratio of Ni in Inconel alloys gives resistance to oxidation and this gives resistance to the formation of oxide layer too. The part of Ni is maximum in case of sample 2 which was exposed to KOH and it is minimum in case of sample 5 which was exposed to HCl. Cr is the element which gives strength to the material to resist corrosion and it is clearly understood from the table-3 that fraction of Cr decreases in every case as compared to the untreated sample. This shows that every sample corroded to some extent and the impact is higher on sample-4 which was exposed to HNO_3.

4.2. Hardness

Fig. 4 shows the variation of micro hardness of different samples which were exposed to different environments.It has been inferred from Vickers hardness test that in every treated sample the value of hardness has reduced in comparison with untreated one (Sample 7), shown in table-4.It is clearly understood by the previous studies that increase in weight percentage of Molybdenum (Mo) and Niobium(Nb) results in the formation of Inter metallic compounds, likely to be Ni_3Nb and $Ni_2(Cr,Mo)$. This process lead to the formation of δ, σ and γ'' phases [24]. It has been observed that these phases increase the micro hardness due to solid-solution strengthening [25]. It is clearly understood from the table 3 that the percentage of Nb has lesser value than the untreated sample value in every case. The maximum percentage of Nbhas been found is 2.7 as shown in table-3 which is also less than untreated sample value. So it could be inferred that the micro-hardness is lesser than untreated sample value in every case.

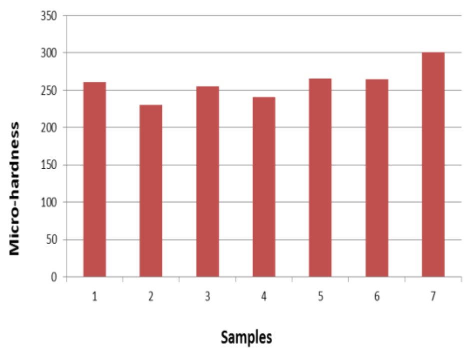


Fig. 4. Variations of microhardness of samples.

As it is already discussed in surface morphology that in case of sample 5 and sample 6 which were exposed to HCl and chromic acid, respectively the impact of corrosion and helium ions irradiation was less hence small number of pores and cavities were observed.The maximum of hardness has been found as 290.35 in case of sample-6 which were exposed to Chromic acid and minimum value has been found as 239.58 in case of sample-2 which is exposed to KOH as shown in Table 4.

Table 4.Micro hardness of the samples.

Sample No.	Microhardness (in HV)
1	275.46
2	239.58
3	284.48
4	252.96
5	277.82
6	290.35
7 (Untreated sample)	301

5. Conclusion

Inconel-625 alloy samples were corroded with six different solutions i.e. H_2SO_4, KOH, NaOH, HNO_3, HCl, and Chromic Acid for the duration of 240 hrs and then helium ion irradiation wasperformed on the samples with the fluence of 10^{15}ions/cm^2. The effect on surface morphology and ratio of elemental constituents have been studied using SEM-EDAX (Scanning electron Microscope-Energy Dispersive X-Ray).The hardness of the samples has been studied using Vickers hardness test.For all the samples helium ions irradiation enhances intergranular corrosion. But in case of sample 5 and sample 6 which were exposed to HCl and chromic acid respectively the impact was less hence small number of pores and cavities were observed. Severe impact has been observed in case of sample of 2 and 3 which were exposed to KOH and NaOH respectively and large numbers of cavities were observed on the surface.

EDAX showed that percentage elemental composition varies in every case and depletion of chromium and niobium has been observed in every case which results in corrosion of samples and decrease in micro hardness too as compared to untreated sample. Hardness decreased in every case as corrosion reduced the percentage of Niobium. As already discussed Sample no 5 and 6 which are exposed to HCl and chromic acid respectively has least affected samples as it comes to defects which are ultimately results of corrosion and ion-irradiation. Sample no-6 which was corroded in chromic acid has maximum fraction of Niobium as 2.7 % and it showed the maximum hardness as compared all other samples.

References

[1] D.L. Graver, in D. Benjamin and C.W. Kirkpatrick (eds.), Metals Handbook; Properties and Selection] Staiidess Steels, Tool Materials and Special Purpose Mttals, 9th edn.,**3**, ASM, Metals Park, OH, 171 (1980).
[2] D. LeBlanc, Nucl. Eng. Des. **240**, (2010) 1644.
[3] Y. C. Sharma, S. Sharma, PortugaliaeElectrochimicaActa**34**(6), 365 (2016).
[4] S. Sharma, Y. C. Sharma, PortugaliaeElectrochimicaActa**37**(1), 1 (2019).
[5] R. Kumar, D. Bhardwaj, Y. C. Sharma, Journal on Material Science**6**(1), 31 (2018).
[6] Y. C. Sharma, R. Kumar, V. V. Sagar, D. Bhardwaj Materials Research Express **6**(2), 026559 (2019).
[7] R. Kumar, Y. C. Sharma, V. V. Sagar, D. Bhardwaj, Iranian Journal of Materials Science and Engineering **17**(2), 20 (2020).
[8] R. Kumar, Y. C. Sharma, V. V. Sagar, D. Bhardwaj, AIP Conference Proceedings **2115**, 030282 (2019).
[9] K.L. Murty, I. Charit, J. Nucl. Mater. **383**, 189 (2008).
[10] G. Velişa, M.W. Ullah, H. Xue, K. Jin, M.L. Crespillo, H. Bei, W.J. William, Y. Zhang, Acta Mater. **135**,54 (2017).
[11] T.N. Yang, C. Lu, G. Velisa, K. Jin, P. Xiu, M.L. Crespillo, Y. Zhang, H. Bei, L. Wang, Acta Mater. **151**,159 (2018).
[12] C.D. Judge, V. Bhakhri, Z. Jiao, R.J. Klassen, G. Was, G.A. Botton, M. Griffiths, J. Nucl. Mater. **492**, 213 (2017).
[13] S.X. Jin, L.P. Guo, Z. Yang, D.J. Fu, C.S. Liu, W. Xiao, R. Tang, F.H. Liu, Y.X. Qiao, Nucl. Inst.Methods B **269**(3),209 (2011).
[14] C. Lu, T. Yang, K. Jin, N. Gao, P. Xiu, Y. Zhang, F. Gao, H. Bei, W.J. William, K. Sun, Y. Dong, Acta Mater. **127**,98 (2017).
[15] K. Jin, W. Guo, C. Lu, M.W. Ullah, Y. Zhang, W.J. Weber, L. Wang, J.D. Poplawsky, H. Bei, Acta Mater. **121**,365 (2016).
[16] C.D. Judge, N. Gauquelin, L. Walters, M. Wright, J.I. Cole, J. Madden, G.A. Botton, M. Griffiths, J. Nucl. Mater. **457**,165 (2015).
[17] H. Zhu, R. Holmes, T. Hanley, J. Davis, K. Short, L. Edwards, Corros. Sci. **91**,1 (2015).
[18] Y. Dai, G.R. Odette, T. Yamamoto, Comprehensive Nuclear Materials, 141 (2012).
[19] P.J. Goodhew, Mater. Sci. Tech. **6**, 950 (1990).

[20] G.S. Was, Fundaments of Radiation Materials Science: Metals and Alloys, Springer-Verlag, Berlin, 2007.
[21] H. Trinkaus, Radiat. Eff. Defects Solids **78**, 189 (1983).
[22] P.J. Goodhew, S.K. Tyler, Proc. Royal Soc. London Ser. A – Math. Phys. Eng. Sci. 377, 151 (1981).
[23] B. Hu, H. Kinoshita, T. Shibayama, H. Takahashi, Mater. Trans. **43**, (6222002).
[24] G.P. Dinda, A.K. Dasgupta, J. Mazumdar, Material Science and Engineering **509**(1-2), 98(2009).
[25] Y.J. Zhang, Y.J. Huang, L. Yang, J.G. Li, Journal of alloys and compounds **570**,70(2013).