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Surface and depth distribution of components in high alloy stainless steel using secondary ion mass spectrometry

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The direct imaging and depth sensitive analysis capabilities of secondary ion mass spectrometry were effectively utilized for surface and depth distribution analysis of major and trace elements (C, Si, Cr, Mn, Fe, Ni, Co and Mo) in high alloy stainless steel sample. Oxygen (O_2^+) primary ion beam with positive secondary ion detection mode was selected for surface and depth distribution analysis of elements in stainless steel sample. Qualitative surface distributions of the elements were obtained by area line scan analysis constructed from surface ion distribution images. 3D analyses were also carried out using O_2^+ primary ion beam in order to investigate spatial distribution of the elements. Surface ion distribution images, 3D analysis and area line scan analysis confirmed the non homogenous spatial distribution of Si and C while other elements Cr, Mn, Fe, Ni, Co and Mo were homogenously distributed over the surface as well as with respect to depth. The non homogenous spatial distribution of Si and C may be due to formation of carbide or precipitates of Si and C. Depth distribution analysis showed uniform depth distribution of all the elements including C and Si within the analysis area of 150 μm × 150 μm.

1. Introduction

Secondary ion mass spectrometry (SIMS) is an extremely sensitive analytical technique, with detection limits in the range of ppm to ppb (for some elements even in the range of ppt), commonly employed for surface and depth distribution analysis of elemental and molecular ions in the sample [1,2]. SIMS is also capable of detecting low atomic number elements including hydrogen [2]. Several advantages offered by SIMS over other surface analysis techniques (RBS, XPS, AES etc.) include the capability for analysing full mass spectrum, very low detection limits, 2D (2-Dimentional) as well as 3D (3-Dimensional) distribution analysis of elemental and molecular ions, high mass resolution (m/dm ~10,000), large dynamic range, direct solid sample analysis, good lateral resolution (~micrometer) for surface imaging and excellent depth resolution (~nm) for depth profile analysis etc [3]. These unique capabilities have popularised the application of SIMS in different fields of science and technology such as semiconductor [3], biological [4], thin films [5], nuclear industry [6] etc.

The fundamental operating principle of SIMS is based on bombarding the sample surface with a beam of energetic primary ions (energies in the range from few eV to keV) followed by the detection of sputtered secondary ions using a mass spectrometer [1]. Most SIMS instruments operate in two modes namely static mode and dynamic mode. In static mode, also known as imaging SIMS mode, the impact energy as well as intensity of primary ion beam are sufficiently low, which provides surface distribution or mapping of atomic or molecular ions with a lateral resolution in the range of micrometer (primarily depending upon the beam size). On the other hand, in case of dynamic mode, the impact energies as well as primary ion beam intensity are relatively high, resulting in continuous sputtering of the sample surface, which is commonly employed for depth distribution studies [7]. Two most commonly used primary ions beams for sputtering the sample surface includes Cesium (Cs^+) and Oxygen (O_2^+) beams. The Cs^+ beam enhances negative ion yield, while O_2^+ beam enhances positive ion yield [8]. This enhancement in the positive and negative secondary ion yields makes SIMS an extremely sensitive technique having detection limits in the range of ppm to ppb, and for some elements even in the range of ppt [2].

Steel is one of the most commonly used structural material in the modern industrial regime. Different types of steels were developed over the last few decades for several applications which include stainless steel, low alloy steel, high alloy steel (HAS) etc. [9]. HAS as a structural material has potential applications in nuclear reactor power plants due to its various advantages such as hardness,

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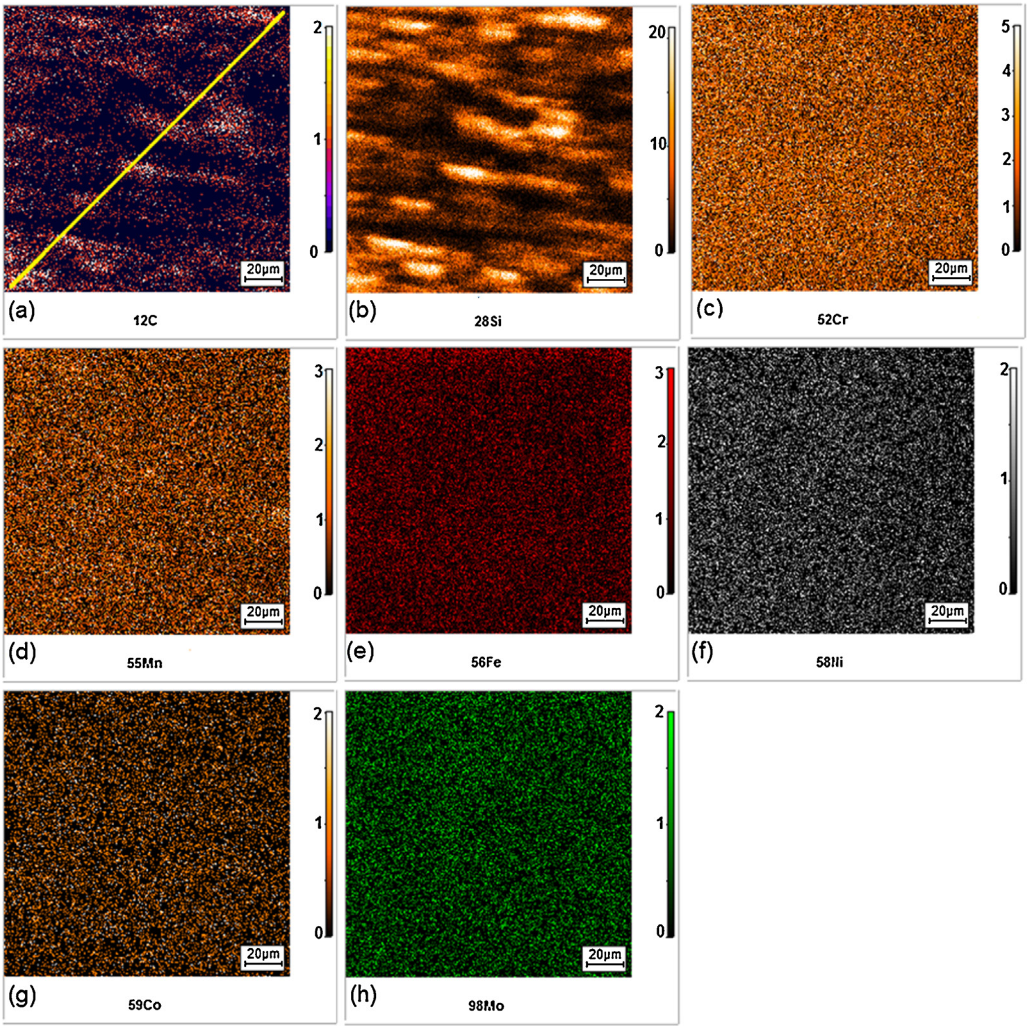

Fig. 1. SIMS images of (a) $^{12}C^+$ (b) $^{28}Si^+$ (c) $^{52}Cr^+$ (d) $^{55}Mn^+$ (e) $^{56}Fe^+$ (f) $^{58}Ni^+$ (g) $^{59}Co+$ and (h) $^{98}Mo^+$, the line in Fig. 1 (a) represents the area line scan region selected for surface distribution studies.

Table 1
Certified composition of the high alloy stainless steel sample.

Sample	Elemental concentration (wt.%)							
	C	Mn	Si	Ni	Cr	Mo	Co	Fe
C4	0.019	1.4	0.27	10.2	18.46	0.265	0.116	69.27

machinability, ductility, high temperature corrosion resistance, good thermal and electric conductivity etc. [9]. Some of the examples of the HAS used in nuclear industries are Sandvick Sweden HT9, Sandvick Sweden HT7, AISI 403, AISI 410, French R8, French EM12 etc. [9].

SIMS is a very useful analytical technique for investigating 2D and 3D distribution of elemental and molecular ions in a sample. Investigation of these distributions helps in understanding various phenomenon such as microstructural changes due to corrosion process, grain boundary diffusion, phase determination etc. For instance, Grams et al. investigated the influence of catalyst preparation conditions on the distribution and composition of active phase using high resolution surface ion images of elements as well as molecular secondary ions acquired from support catalyst using SIMS [10]. Krecar et al. investigated 2D and 3D distribution of the sintering activator of phosphorus and carbon in steel by means of SIMS [11]. They have concluded from the obtained ion distribution patterns that with increase in phosphorous concentration in steel, phosphorous gets precipitated in the grain boundary regions, especially at higher sintering temperatures [11]. Gammer et al. utilized SIMS for 3D distribution studies of main and trace components in aluminium alloyed High Speed Steel (HSS) [12]. They have compared the distribution of these elements in HSS and aluminium alloyed HSS and found that the concentration of Al influences the distribution of Si, Ti, K, Ca and Na in HSS [12]. Rossi et al. utilized three surface analytical techniques XPS, AES and ToF-SIMS to characterize the surface films formed on stainless steels following mechanical polishing and immersion in an aggressive 6% $FeCl_3$ solution, for testing the pitting corrosion resistance of stainless steel [13]. The reported literature suggests the importance of determining the surface microstructure and depth distribution analysis of elements in steel, not only during their production stage but also during the life time of material for monitoring as well as to understand the degradation phenomena. The quantitative analysis in steel samples has been studied extensively using SIMS [14]. Since very little information regarding the surface and depth distribution studies of composite elements in high alloy stainless steel sample using SIMS has been reported. Hence, it was considered worthwhile to study this for furthering the understanding of the processes in HAS.

In this work, surface and depth distribution analysis of composite elements C, Si, Cr, Mn, Fe, Ni, Co and Mo in high alloy stainless steel (HAS) samples were investigated using SIMS. All the anal-