Letters

Nitrogen analysis in an electron beam welded austenitic stainless steel, Nitronic 33

TABLE I Chemical analysis of Nitronic 33

Element	Wt %
C	0.041
Mn	12.39
P	0.019
S	0.005
Si	0.50
Cr	17.92
Ni	3.11
N	0.33
Mo	0.19

It has been shown that the electron beam (EB) weldability of high-nitrogen, high-manganese, austenitic stainless steels is directly related to nitrogen content [1, 2]. High nitrogen concentrations can cause weld-metal eruptions, resulting in excessive splatter, concavity, and porosity. Weld puddle quiescence in Nitronic 33 (18 wt% Cr–3 wt% Ni–13 wt% Mn–0.3 wt% N) is difficult to achieve unless the weld parameters, such as welding current and travel speed, are carefully balanced and controlled. This difficulty suggests excessive nitrogen evolution [3]. Nitrogen losses of between 10 and 15% in Nitronic 40 (21 wt% Cr–6 wt% Ni–9 wt% Mn–0.3 wt% N), another high nitrogen austenitic stainless steel, were observed in single-pass EB welds on 6 mm plate [1]. Since the nitrogen addition to Nitronic 33 is mainly to provide interstitial solid solution strengthening, a serious concern is the possibility of weld zone nitrogen depletion which can occur as a result of EB welding. The purpose of this work was to measure the nitrogen concentration and distribution in EB welded Nitronic 33 to determine the extent of nitrogen depletion and its relationship to weld mechanical properties.

The Nitronic 33 material used in this work, was 18 mm (0.750 in) thick, mill annealed plate, characterized by a uniform and equiaxed microstructure typical of austenitic stainless steels. The bulk chemical analysis is listed in Table I.

EB butt welds were made with the following parameters: 35 kV, 170 mA, 02.5 m min^{-1} (10 in min^{-1}) and 0.2 (8 in) gun-to-work distance; the beam focus was well above the workpiece surface. There were no cracks, pores, or other anomalies observed in the weld specimens examined; the weld microstructure was considered to be typical of austenitic stainless steels. A microhardness survey showed that the hardness of all regions of the weld and base metal was relatively uniform.

The localized measurement of nitrogen in an EB weld of Nitronic 33 uses a method based on nuclear reaction analysis. In this technique, the surface region of interest is bombarded with high energy deuterons which interact with the nitrogen present producing a spectral distribution of protons whose yield is proportional to the nitrogen concentration. The nuclear probe is a nondestructive method of measuring nitrogen in localized regions compared to the relatively larger material volumes required for conventional interstitial element analysis. Nitrogen concentrations were measured

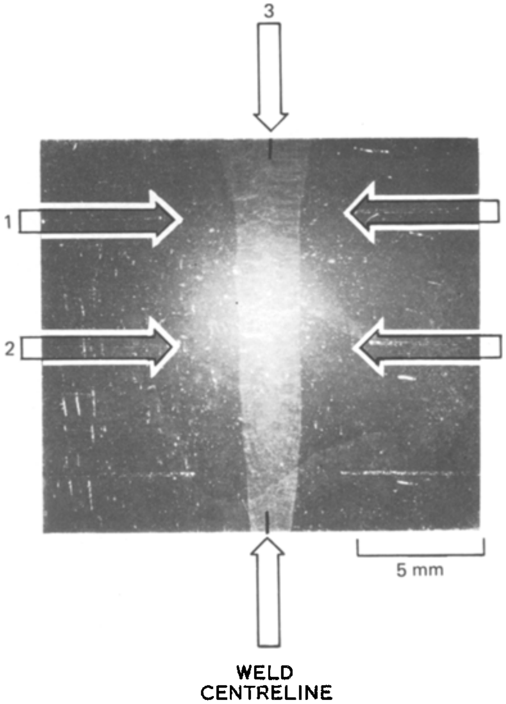

Figure 1 Macrograph of transverse section from Nitronic 33 EB weld, showing locations of regions selected for nitrogen concentration profiling.

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in three regions of a transverse weld section: two were made by scanning across both the base metal and the weld metal, and the third by scanning along the weld centreline (see Fig. 1). Specimens were probed using a 0.3 mm x 0.3 mm beam size; the data represent the average concentration within a 3 μm thick layer, starting 0.25 μm below the surface to avoid spurious surface effects.

TABLE II Average nitrogen concentrations in Nitronic 33

Region	Nitrogen concentration (ppm)
Base metal	3730 ± 125
Fusion line	3470 ± 80
Weld metal at centerline	3310 ± 90
Weld metal adjacent to centreline	3310 ± 115

The results of the nitrogen analyses, shown in Fig. 2, indicate that a loss of nitrogen in the weld metal occurred as a result of the single-pass EB weld. The nitrogen concentration in the weld, including the centreline and adjacent weld metal, is relatively uniform (i.e., ≈ 3300 ppm). Slightly higher nitrogen concentrations were measured at the "fusion line" (i.e., the transition zone between weld and base metal). The concentrations measured at the fusion line approximate the value of the average concentration taken between the weld metal and the base metal. A compositional transition of this type would be expected in this region of the weld but the spatial resolution of these measurements was not fine enough to determine the slope of the concentration gradient through this region. The average nitrogen concentrations for this specimen are given in Table II.

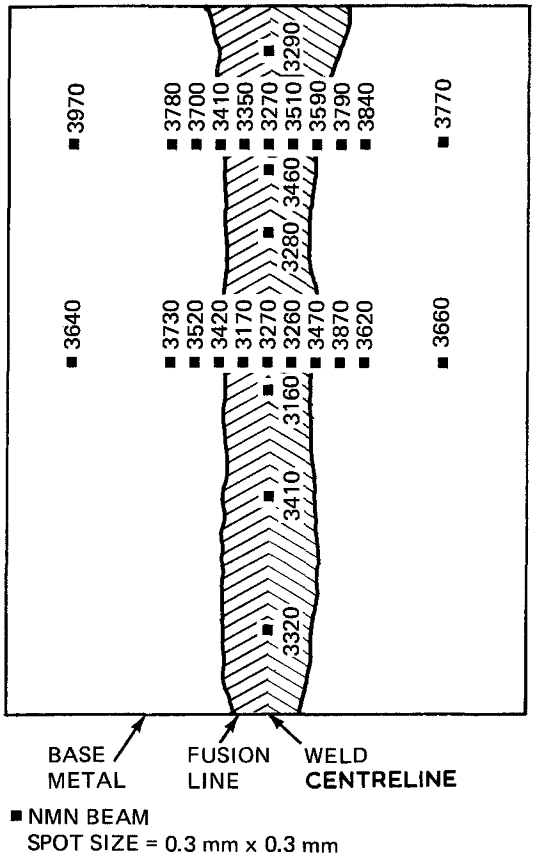

Figure 2 Nitrogen concentrations (wt ppm) in regions of EB welded Nitronic 33.

A comparison of nitrogen concentrations between the weld and base metal shows that the nitrogen loss was approximately 11 wt% as a result of welding. This depletion of nitrogen is similar to the 10 to 15 wt% decrease observed by Brooks [1] in single-pass EB welding of Nitronic 40 in a plate having one third the thickness. Thus it would appear that nitrogen losses of this order would be expected when single-pass EB welding is conducted on high nitrogen austenitic stainless steel plate under similar conditions (i.e., when the metal is molten for approximately 0.1 sec under a pressure of 10^{-4} torr).

The nitrogen loss in the Nitronic 33 EB weld had no apparent negative effect on weld mechanical properties, including tensile, impact, and fatigue strengths; in fact, the Charpy impact resistance was greatly improved. These data are shown in Table III. It can be concluded that nitrogen depletion of the order of 10 wt% occurs as a result of EB welding Nitronic 33 but does not lead to significant mechanical property degradation of the weld metal.

TABLE III Comparison of mechanical properties in EB welded Nitronic 33

	Weld*	Base metal*
Ultimate tensile strength (MPa)	742	728
Yield strength (MPa)	424	404
Elongation (%)	54	60
Charpy impact (J)	358	262
Fatigue endurance limit (MPa)	396	393

* [3].

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X-ray Debye temperature of sodium chlorate

The Debye temperature plays an important role in the study of a large number of solid-state problems involving lattice vibrations. A number of physical parameters such as mean square atomic displacements, Herbstein [1], and elastic constants, Gazzara and Middleton [2], are known to depend upon the Debye temperature of a solid. It has been shown by Salter, [3], that Debye temperatures obtained by different physical properties will not, in general, be equal. The survey of the literature shows that there is a scarcity of adequate data on X-ray Debye temperatures for the isomorphic structures of sodium bromate ($NaBrO_3$) and sodium chlorate ($NaClO_3$). The X-ray Debye temperature of sodium bromate has been determined by Kulkanri and Wathore [4] but, to our knowledge, no results have yet been published on the X-ray Debye temperature of sodium chlorate.

In the present work, measurements of X-ray diffracted intensities at various temperatures were carried out with the object of extracting X-ray Debye temperature θ_M for sodium chlorate. The θ_M obtained in the present investigation was compared with the Debye temperature obtained from elastic constants, Mason [5].

A method which depends on the principle of measuring the integrated intensities of a large number of Bragg reflections at a fixed temperature was first outlined by Buerger [6]. This method has been described in detail by Walford and Schoeffel [7] and has been used here to determine the X-ray Debye temperature θ_M. Recently Kulkarni and Bichile [8] used this method to obtain X-ray Debye temperatures of $Ba(NO_3)_2$ assuming a single Debye–Waller factor (M) for all three atoms in the compound.

The average vibrational amplitudes are related to the Bragg intensities, within the quasi-harmonic approximation, through the Debye–Waller theory. The integrated intensity, I, from a cubic powder sample can be expressed as follows, James [9]

$$I = KL_pP|F|^2, \qquad (1)$$

where K is a constant; L_p is a function of the Bragg angle known as the Lorentz-polarization factor; P is multiplicity; and $|F|$ is the modulus of the structure factor.

The structure factor for sodium chlorate (space group T^4) can be written as

$$F(h\,k\,l) = f_{Na}F_{Na}e^{-M_{Na}} + f_{cl}F_{cl}e^{-M_{cl}} + 3f_OF_Oe^{-M_O}. \qquad (2)$$

The exponential terms in Equation 2 represent the Debye–Waller factors for the three constituent atoms of sodium, chlorine and oxygen; f_{Na}, f_{Cl} and f_O are their respective atomic scattering factors; F_{Na}, F_{Cl} and F_O are their respective structure factors which are sine and cosine functions of hkl values; all other terms have standard meanings (as defined in the International Tables for crystallography, 1969, [10]). According to James [9], the Debye–Waller factor is defined as

$$M(T) = \frac{6h^2T}{mK\theta_M^2}\left\{\phi(x) + \frac{x}{4}\right\}\frac{\sin^2\theta}{\lambda^2} \qquad (3)$$