J. Indian Inst. Sci., 66, June 1986, pp. 385–395

Influence of processing conditions on the properties of investment cast austenitic stainless steel

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Received on June 10, 1986.

Abstract

A full factorial 2^3 matrix was designed to study the effect of simultaneous variations in the pressure of the melting and casting environment, casting size and mould preheating temperature on the mechanical behaviour and intergranular corrosion of an investment cast austenitic stainless steel. The results indicate that the pressure of the melting and casting environment exerts the most significant influence on the properties examined. Combinations of the variables which give rise to improved properties have been identified.

Key words: Stainless steel, vacuum melting and casting, mechanical properties, corrosion, investment casting, design of experiments, austenite, delta ferrite.

1. Introduction

Austenitic stainless steel parts are used in applications requiring good mechanical behaviour and corrosion resistance. Investment casting of this alloy would lead to additional advantages of good surface finish and close dimensional tolerances. However, several questions need to be answered before the operating conditions for this process are chosen. For instance, it is broadly known that melting and casting of alloys in vacuum results in a reduction of the detrimental effects of gases[1–5], a reduction in impurities through volatalization[2,4,6] and facilitates carbon removal[7]. But what needs to be examined is whether applying vacuum melting and casting to investment cast austenitic stainless steel would result in any significant improvement in properties which would justify the additional expenditure. Also it is customary in investment casting practice to preheat the mould to a high temperature to aid mould filling. It needs to be ascertained whether the slow cooling rates brought about by high preheating temperatures, especially through the sensitization range, would adversely affect the resistance to intergranular corrosion, even when the carbon content is low as with vacuum melted and cast samples. Any influence of such slow cooling on the mechanical properties also needs careful examination. Similarly, the effect of casting size on the properties also needs proper understanding. Design of experiments can provide elegant answers to these questions with a minimum number of trials[8] and provides for simultaneous variations in the experimental variables chosen. In the present paper, a full factorial 2^3 matrix has been chosen to study the effect of the pressure of the melting and casting environment, mould preheating temperature and casting size upon the mechanical behaviour (ultimate tensile strength and elongation) and intergranular corrosion rate of an investment cast austenitic stainless steel. The significance of each variable on these properties has been

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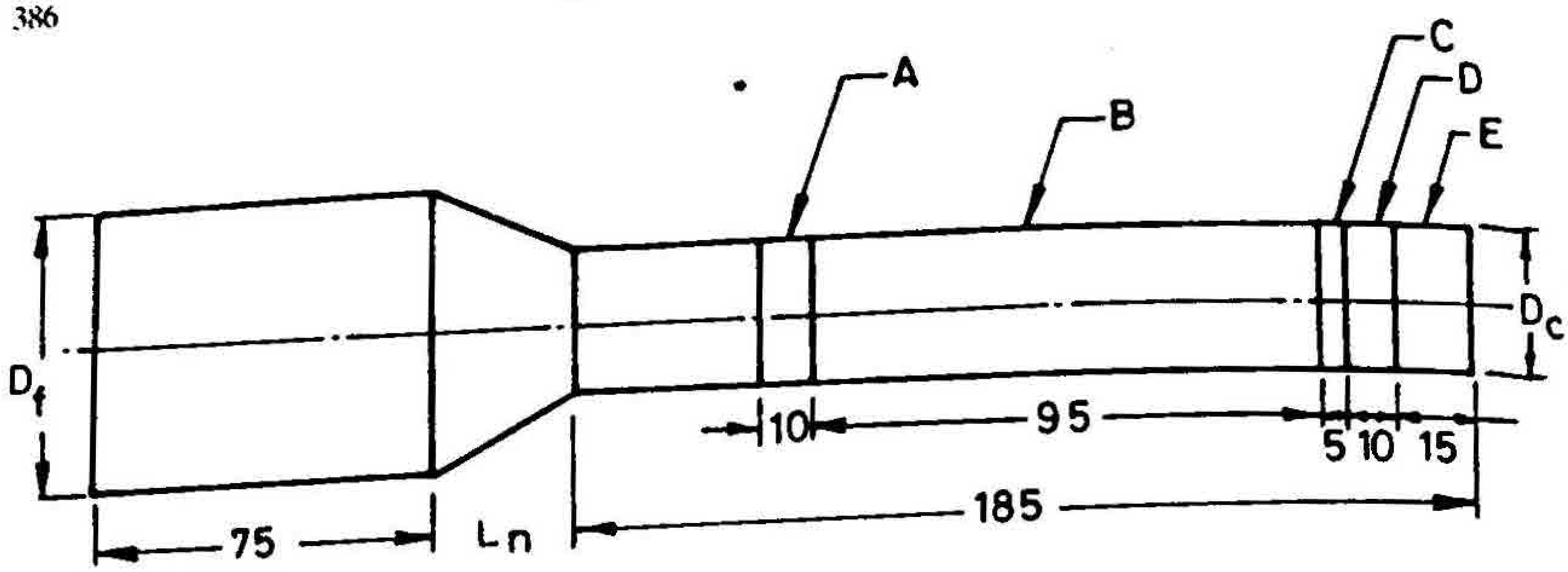


FIG. 1. Details showing location of test specimens in the test casting taken for various tests.
D_f = Feeder diameter; D_c = Casting diameter
For locations A, B, C and D tests conducted are macrostructure, mechanical properties, intergranular corrosion and microstructure respectively.

assessed and regression equations relating each property to the variables have been developed.

2. Experimental procedure

Alloy studied: All experiments were carried out with stainless steel having the following composition.

Chromium: 18.2%, Nickel: 7.9%, Carbon: 0.08%
Silicon: 0.8%, Manganese: 0.6%, Copper: Nil
Titanium: Nil, Nitrogen: 0.049%, Phosphorus: 0.04%
Iron: Bal.

Melting: The alloy was air melted in an 8000 HZ, 30 KW, 20 lb capacity Brown–Boveri induction furnace. For vacuum melting and casting, an 8000 HZ, 12.5 KW, 17 lb capacity GCA vacuum induction furnace provided with a vacuum system capable of reaching 3×10^{-5} Torr, was employed. The melting stock consisted of 60 mm dia austenitic stainless steel bars and no additions were made to the melt in all cases.

Test casting: Cylindrical bar castings (fig. 1) were cast under various conditions. Specimens were machined out at locations indicated in fig. 1, and the tensile and the intergranular corrosion behaviour were studied.

Moulds: Investment shells were prepared by using the shell making process developed in the foundry laboratory of the Indian Institute of Science[9,10]. Moulds were preheated inside a resistance heating furnace.

Tests on casting:

a) *Tension test*: ASTM standard specimens were machined from the mid-portion of test casting and the ultimate tensile strength and percentage elongation were determined.

b) *Intergranular corrosion*: Circular disc samples were machined from bottom portion of test castings. They were abraded using water emery (first 400 and next 600 size) till all machining marks were removed. Corrosion test was carried out by following ASTM standard No.A.262 practice B: ferric sulphate-sulphuric acid method.

Statistically designed experiments:

Selected levels of the variables are shown in Table I. The upper and lower levels were so selected as to allow the determination of the influence of the variables over a wide range, consistent with the ease of maintaining them under experimental conditions. The zero levels correspond to the arithmetic mean of the upper and lower levels.

Details of the full factorial 2^3 matrix are indicated in Table II. Two trials were made for each treatment to determine: a) the adequacy of both the linear and the non-linear models obtained by regression analysis of the results, and b) the significance of the regression coefficients.

3. Results and discussion

The experimental design and the regression analyses are effected assuming the following hypotheses (Table II):

a) The optimization parameter Y (here UTS, % elongation and intergranular corrosion) is a random population normally distributed,

Table I
Levels of variables

Level	x_1 °C	x_2 mm Hg	x_3 Dia (mm)
Upper (+)	600	760	40
Lower (−)	25	0.25	15

Table II
Results of statistical design of experiments

Treatment no.	x_1 (Mould temp.)	x_2 (Melt Pr.)	x_3 (Casting size)	Ultimate tensile strength MN/m^2		% Elongation		Intergranular corrosion in/month $\times 10^{-3}$	
				Trial 1	Trial 2	Trial 1	Trial 2	Trial 1	Trial 2
1	+	+	+	357.3	350.2	25.5	23.1	7.5943	7.7025
2	+	−	+	505.0	520.5	48.9	50.6	3.8741	3.9841
3	+	+	−	345.2	339.8	21.2	20.3	6.1067	5.9830
4	+	−	−	461.2	467.6	25.4	26.9	3.7647	3.8444
5	−	+	+	375.2	361.5	24.3	22.1	4.5515	4.6886
6	−	−	+	565.6	558.9	32.5	31.4	3.7691	3.8705
7	−	+	−	320.4	314.5	20.3	19.0	4.4826	4.5204
8	−	−	−	499.8	511.7	39.9	41.6	3.6974	3.7596

b) the variance of Y does not depend on its absolute value,

c) a linear mathematical model is suitable for the factors considered.

Variance of reproducibility

Using the definition[8]:

$$S^2(Y) = \sum_1^N \frac{n(Y_{ig} - Y_i)^2}{N(n-1)}. \tag{1}$$

The variance of reproducibility was calculated for each parameter. The values are found to be 48.11, 1.39 and 0.5×10^{-2} for ultimate tensile strength, % elongation and intergranular corrosion respectively. The deviation from the mean was less than 10% in all treatments.

Homogeneity of variance

Using Cochran's Criterion[8], which is the ratio of the maximum variance of one of the eight treatments to the sum of all the variance for every treatment, *viz.*,

$$G = \frac{S^2 \max}{\sum_1^N S_t^2} \tag{2}$$

where

$$S_t^2 = \frac{\sum_1^n (Y_{ig} - Y_i)^2}{n-1} \tag{3}$$

The experimental Cochran's ratios for ultimate tensile strength, % elongation and intergranular corrosion are 0.312, 0.258 and 0.236 respectively. All these values are less than 0.69, the tabulated value for 2^3 matrix with two replications of each treatment. Thus, the homogeneity of variance for each parameter noted above is confirmed and the prerequisite for the application of regression analysis of these experimental results is satisfied.

Regression analysis

It is assumed that the relation between the optimization parameter and the variables can be expressed in the form:

$$Y = a_0 + a_1x_1 + a_2x_2 + a_3x_3. \tag{4}$$

Using the least squares method for an orthogonal matrix, as employed in this investigation, the regression coefficients may be evaluated as:

$$a_0 = \frac{\sum_1^N Y_i}{N}, \tag{5}$$

$$a_1 = \frac{\sum_1^N Y_i x_1}{N}, \tag{6}$$

$$a_2 = \frac{\sum_1^N Y_i X_{2i}}{N}, \tag{7}$$

$$a_3 = \frac{\sum_1^N Y_i X_{3i}}{N}, \tag{8}$$

where Y_i is the response in the ith treatment.

For the present experimental results the regression equations are:

$$Y_U = 428.39 - 10.04x_1 - 82.90x_2 + 20.86x_3 \tag{9}$$

$$Y_E = 29.562 + 0.675x_1 - 7.59x_2 + 2.74x_3 \tag{10}$$

$$Y_{IC} = (4.762 + 0.595x_1 + 0.942x_2 + 0.242x_3)\ 10^{-3} \tag{11}$$

for ultimate tensile strength, % elongation and intergranular corrosion respectively.

Adequacy of linear model

This may be confirmed by Fisher's ratio[8]

$$F = \frac{S_{ad}^2}{S_y}$$

where S_{ad}^2 = variance of adequacy given by

$$S_{ad}^2 = \sum_1^N n \frac{(\overline{Y}_i - Y_i)^2}{f}$$

where f is the degree of freedom given by $N-(k+1)$ for a 2^k matrix. Here $N = 8$ and $k = 3$.

The experimental Fisher's ratio for ultimate tensile strength, % elongation and intergranular corrosion are found to be 19.38, 96.68 and 324.58 respectively. All these are higher than 6.09 which is the F ratio tabulated at 5% significant level for the degree of freedom of 4 at 7 and 4 for mean squares of error and class respectively. Thus, a linear regression model is inadequate in all the cases considered.

Regression model with interaction of variables

It was, therefore, decided to examine if a non-linear model of the form:

$$Y = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + a_4x_1x_2 + a_5x_2x_3 + a_6x_3x_1 + a_7x_1x_2x_3 \tag{14}$$

would be adequate instead of a linear model.

The regression equations thus obtained were:

$$Y_U = 428.39 - 10.04x_1 - 82.90x_2 + 20.86x_3 + 12.68x_1x_2 - 5.35x_2x_3 - 5.96x_3x_1 - 3.93x_1x_2x_3, \quad (15)$$

$$Y_E = 29.562 + 0.675x_1 - 7.59x_2 + 2.74x_3 - 0.125x_1x_2 - 0.963x_2x_3 + 4.05x_3x_1 - 4.05x_1x_2x_3, \quad (16)$$

$$Y_{IC} = (4.762 + 0.595x_1 + 0.942x_2 + 0.242x_3 + 0.548x_1x_2 + 0.188x_2x_3 - 0.190x_3x_1 + 0.182x_1x_2x_3)10^{-3}. \quad (17)$$

By similar analysis the experimental F ratios of the new model for ultimate tensile strength % elongation and intergranular corrosion are respectively 4.16×10^{-6}, 0.204×10^{-11} and 0.118×10^{-10}. These values are much lower than the tabulated Fisher's ratio (6.09). Thus, this model is adequate for all the three parameters.

Significance of the regression coefficients in the new model

The confidence interval Δ_{bj}, for a given parameter may be written as

$$\Delta_{bj} = \frac{tS(y)}{\sqrt{N}} \quad (18)$$

where t is the student's t at a 5% significance level, and $S(y)$ is the square root of the variance of reproducibility. The values of Δ_{bj} are calculated as 5.66, 0.96 and 0.06 for ultimate tensile strength, percentage elongation and intergranular corrosion respective-

Table III
Significance of coefficients

Item	UTS	% Elongation	Intergranula corrosion
Adequacy of model	Yes	Yes	Yes
Δ_{bj}	5.66	0.96	0.06
Coefficient of x_1, a_1	−10.04	0.675	0.06
Significance	Yes	No	Yes
Coefficient of x_2, a_2	−82.9	−7.59	0.091
Significance	Yes	Yes	Yes
Coefficient of x_3, a_3	20.86	2.74	0.02
Significance	Yes	Yes	No
Coefficient of x_1x_2, a_4	12.68	−0.125	0.055
Significance	Yes	No	No
Coefficient of x_2x_3, a_5	−5.35	−963	0.02
Significance	No	Yes	No
Coefficient of x_3x_1, a_6	−5.96	4.05	0.02
Significance	Yes	Yes	No
Coefficient of $x_1x_2x_3$, a_7	−3.93	4.05	0.02
Significance	No	Yes	No

ly. The significance of the coefficients in the new model is decided from coefficient Δ_{b_j} (Table III).

Retaining the significant coefficients only (using Table III) the final regression equations for the properties considered are:

a) Ultimate tensile strength

$$U = 428.39 - 10.04x_1 - 82.9x_2 + 20.33x_3 + 12.68x_1x_2 - 5.96x_3x_1. \tag{19}$$

b) Percentage elongation

$$E = 29.56 - 7.59x_2 + 2.74x_3 - 0.96x_2x_3 + 4.05x_3x_1 - 4.05x_1x_2x_3. \tag{20}$$

c) Intergranular corrosion

$$I = (4.76 + 0.59x_1 + 0.94x_2 + 0.24x_3 + 0.55x_1x_2 + 0.19x_2x_3 + 0.19x_3x_1 + 0.18x_1x_2x_3)\ 10^{-3}. \tag{21}$$

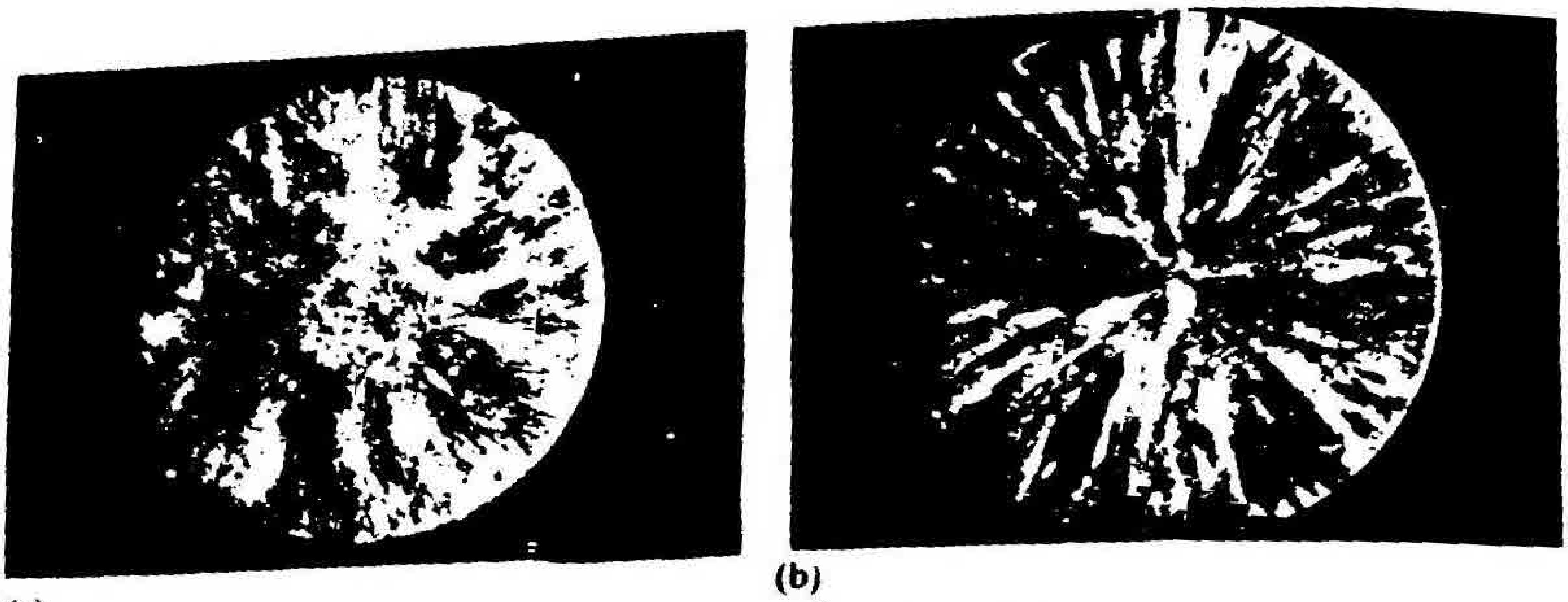
In what follows the relation between each property and the variables will be examined and analysed.

Ultimate tensile strength

It is seen from equation 19 that the pressure of melting and casting environment is the most significant factor affecting ultimate tensile strength. The macrostructure[11,12] and microstructure[13,14] mainly contribute to the ultimate tensile strength behaviour. The macrostructure of vacuum melted and cast samples displayed a columnar equiaxed transition zone towards the centre [Type A + Type B solidification[11]] as against a predominantly columnar structure showed by air melted and cast samples [Type B solidification[11]]. The observed macrostructures agree well with those predicted by superimposing the computed values of nickel and chromium equivalents (Table IV) on

Table IV
Composition of stainless steel

Element	% composition of air melt	Vacuum melt
Chromium	16.00	17.00
Nickel	7.90	7.90
Carbon	0.08	0.04
Silicon	0.08	0.08
Sulphur	0.04	0.04
Phosphorus	0.04	0.04
Manganese	0.60	0.60
Nitrogen	0.049	0.035
Iron	Bal	Bal
Nickel equivalent	11.60	10.10
Chromium equivalent	17.75	19.00



(a) (b)

FIG. 2. Typical macrostructures. (a) Vacuum melt; (b) Air melt.

the composition-structure diagram constructed by McTighe and Beech[11]. Considering the role of macrostructure the presence of fine equiaxed grains in the central region, from which tensile specimens were machined for the present investigation, could lead to high tensile strength. As seen in the study all vacuum-melted (fig. 2) and cast samples of austenitic stainless steel often display a columnar equiaxed transition zone towards the centre [Type A + Type B solidification[11]] as against predominantly columnar grains displayed by air-melted (fig. 2) and cast samples. It was noted that changes in casting size did not alter the type of solidification for a given melting environment.

Considering the microstructure two factors significantly affect the ultimate tensile strength. They are delta-ferrite content and inclusions content. Delta-ferrite in austenitic stainless steel improves the strength by dispersion strengthening[13,14], while the presence of inclusions may reduce the tensile strength[15]. The vacuum-melted samples show greater amount of delta-ferrite content than air-melted counterparts (Table V). In addition, the amount of inclusions in the latter is more than the former. Thus both the microstructural and macrostructural aspects explain the significance of melting and casting environment upon tensile strength.

Table V
Delta-ferrite content

Treatment no.	x_1 (Mould temp.)	x_2 (Melt pressure)	x_3 (Casting size)	Average delta-ferrite content, %
1	+	+	+	Traces only
2	+	−	+	4.9
3	+	+	−	Not detected
4	+	−	−	4.1
5	−	+	+	Traces only
6	−	−	+	5.1
7	−	+	−	Not detected
8	−	−	−	4.6

Elongation

A study of equation 20 shows that here also the melting and casting environment has the most significant effect upon the elongation.

It is reported that the percentage elongation is inversely proportional to the inclusion content[15]. Irvine *et al*[16] have further shown that the total strain at fracture decreases exponentially with increase in volume of inclusions. Orehoski[1] has demonstrated that vacuum-melted alloys show a considerable reduction in inclusion content in relation to their air-melted counterparts. The results of the present work are consistent with these observations.

Elongation is affected to a greater extent, (150%) than the tensile strength (nearly 75%) when the melting and casting environment is changed from vacuum to air. This also points out towards the stronger role of inclusions in affecting the elongation values.

Intergranular corrosion

It is seen from regression equation 21 that the melting and casting environment again exerts higher influence upon intergranular corrosion. The decrease in intergranular corrosion of vacuum-melted and cast samples may be due to three factors, i) decrease in the amount of carbon content; ii) presence of delta-ferrite; iii) longer grain boundary length available for carbide precipitation.

Lower carbon level leads to a considerable decrease in intergranular corrosion. In vacuum melting the carbon level is typically reduced from 0.08 to 0.04%. In addition, Columbier and Hochman[17] and others[20,21] have shown that the presence of delta-ferrite can also decrease intergranular corrosion.

Investigations also showed[18,22] that with all other factors remaining constant, the size of precipitated carbide would influence the intergranular corrosion. It is shown that with a carbide width of less than 200 microns stainless steels are not susceptible to intergranular corrosion. A major factor that influences the carbide width was shown to be the austenite grain size. It is clear that a finer grain would mean a longer grain boundary available for carbides to precipitate. Hence the precipitate will be finer in fine grain alloys, when compared to coarse grain ones. In the present work most of the vacuum-melted stainless steels had finer grains when compared to their air-melted counterparts. Therefore, this factor might also have contributed to the low intergranular corrosion susceptibility of vacuum-melted samples.

4. Conclusions

The pressure of the melting and casting environment has the most significant effect upon the properties of the austenitic stainless steel castings examined; vacuum melting and casting leads to significant improvements in mechanical behaviour and resistance to intergranular corrosion.

Under vacuum melting and casting conditions, larger casting sizes and lower mould temperatures promote better ultimate tensile strength; however, better elongation is obtained when both the mould temperature and the casting size are at their higher levels. Further, under vacuum melting and casting, resistance to intergranular corrosion increases when both the casting size and mould temperature are at their lower levels. However, the variation in intergranular corrosion rate with casting size or mould temperature is not very high under vacuum melting and casting conditions.

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