The effect of nitrogen additions on the structure and properties of AISI 310S stainless steel

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Synopsis

The object of this study was to investigate the influence of nitrogen on the structure and properties of AISI 310S steel. Experimental ingots of the steel with nitrogen contents of up to 0,525 per cent by weight were prepared, hot-rolled, and tested after various treatments. The rollability of the material was adequate. The yield strength, tensile strength, hardness, and fatigue properties of the new alloys improved with increasing nitrogen content. The impact properties improved slightly. Empirical equations were developed for the calculation of the yield and tensile strengths, as well as the difference between these two properties, with increasing nitrogen content.

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Introduction

Austenitic stainless steels have moderate strength[1]: their yield strength varies between 220 and 320 MPa, and their tensile strength between 500 and 600 MPa. They are more costly than ferritic or martensitic stainless steels owing to their higher nickel content, but their cost can be reduced either by an increase in the strength of the material or by a reduction in nickel content. The strength of stainless steels can be increased by suitable alloying, as by the addition of nitrogen or other elements. The nickel content can be reduced by the addition of elements such as manganese and nitrogen to the steel.

Nitrogen is a strong austenite stabilizer[2], and the yield and tensile strengths of nitrogen-containing steels increase with increasing nitrogen content with no adverse effects on ductility[2-5]. The rate of fatigue-crack growth decreases with increasing nitrogen content[2], while the creep strength is enhanced by the addition of nitrogen[2,6,7].

The study described here was undertaken to examine the influence of nitrogen on the structure and properties of stainless steel type AISI 310S.

Materials and experimental procedures

Twelve 6 kg ingots were produced in a laboratory induction vacuum furnace under a nitrogen atmosphere of 600 mbar. Nitrogen was introduced into the steel by the addition of nitrogenized low-carbon ferrochromium. The chemical analyses of the ingots are shown in Table I. Details of the ingot production have been given elsewhere[8].

The ingots were annealed at 1200°C for 2 hours, hot-rolled to slabs of 15 mm thickness (76 per cent reduction), and quenched in water. The finished rolling temperature was estimated to be between 800 and 900°C. Small pieces cut from the water-quenched slabs were solution heat-treated at 1200°C, followed by quenching in water. From this, the time required for all particles of the precipitate in the steel to be taken into solution was determined. It was established by use of optical metallography that all the precipitates could be taken into solution in 6 hours at 1200°C. Accordingly, all the slabs were heated at 1200°C for 6 hours and then quenched in water. The precipitation characteristics of the samples were determined after they had been heated for 4 hours at temperatures ranging from 800 to 1200°C.

Specimens for Charpy, tensile, and fatigue tests were prepared from the slabs. Charpy-impact tests were carried out on transverse specimens. The maximum recording capacity of the machine was attained with specimens of 100 mm^2 in cross-section taken from the base alloy (first row in Table I). Subsize specimens of 50 mm^2 in cross-section (10 mm deep $\times$ 5 mm thick) were subjected to measurements of the energy absorbed. Longitudinal and transverse tensile specimens were machined from all the ingots after the hot-rolled material had been annealed and tested. Fatigue tests were carried out on 4-point bending-test specimens taken from longitudinal sections of the slabs. The tests were carried out with an MTS servo-hydraulic testing machine of 50 kN capacity. Tests on the growth of fatigue cracks were conducted in air at an R ratio of 0,5 utilizing a sine waveform at a frequency of 20 Hz. The crack extension was measured by means of a low-power travelling microscope. Vickers hardness tests were carried out at a load of 10 kgf and a dwelling time of 5 seconds.

The phases in the alloys were determined by X-ray-diffraction analysis on aged samples and on samples that had been treated at 1200°C for 6 hours. The diffraction patterns were obtained with a computer-controlled vertical goniometer using copper radiation and a graphite monochromator.

Results

Breakdown and thermal processing of ingots

Visual examination of the hot-rolled samples showed that edge and surface cracking had occurred in the samples containing more than 0,15 per cent nitrogen by weight. The influence of nitrogen on the edge-cracking characteristics can be seen from Figure 1. The quality of the edge deteriorated to some extent with increasing nitrogen content.

Table I

Chemical analyses of the ingots (in percentages)

C	S	P	Mn	Si	Cr	Ni	N
0,053*	0,007*	0,026*	1,65*	0,45*	24,70*	19,00*	0,016*
0,045	0,010	0,024	1,65	0,37	25,75	18,89	0,131
0,054	0,007	0,025	1,64	0,44	25,70	18,87	0,153
0,064	0,010	0,029	1,44	0,36	25,20	19,10	0,201
0,048	0,009	0,027	1,46	0,33	25,07	19,09	0,219
0,036	0,011	0,032	1,71	0,35	24,96	19,40	0,268
0,044	0,003	0,027	1,41	0,36	25,63	19,10	0,284
0,040	0,010	0,032	1,68	0,34	25,95	18,85	0,372
0,042	0,004	0,028	1,68	0,41	25,56	18,75	0,404
0,047	0,007	0,028	1,68	0,45	26,26	18,62	0,470
0,043	0,005	0,027	1,66	0,41	25,91	18,59	0,503
0,044	0,005	0,028	1,66	0,40	26,60	18,47	0,525

* Composition of AISI 310S steel before the addition of nitrogen

Hardness of 310S containing nitrogen

Vickers-hardness tests were carried out according to ASTM specification E92-82 using a load of 10 kg. The variations in hardness with nitrogen content for the samples in the as-cast, hot-rolled, and annealed conditions are presented in Figure 2. Statistical analyses showed that hardness increased linearly with nitrogen content. The results of these analyses are shown in Table II.

Figure 2 indicates that the hot-rolled material had the highest hardness. This was probably due to strain hardening of the material and precipitation within it. Strain hardening and precipitation are possible when the temperature of the alloy drops below about 950°C.

The hardness of the aged specimens is plotted against aging temperature in Figure 3. It can be seen that the aging temperature had little effect on the hardness. The hardness of the sample containing 0,530 per cent nitrogen aged at 850°C was well above the values obtained in samples aged at other temperatures because of the presence of pearlite-like regions in the microstructure (Figure 8).

Tensile properties

The effect of nitrogen content on tensile properties is shown in Figure 4. It can be seen that the yield and tensile strengths increased linearly with increasing nitrogen content. This trend was similar for materials of two different grain sizes. A decrease in grain size from 1,34 to 0,66 mm increased the strength significantly. Regression analyses on the data confirmed that the increase in strength was related linearly to the nitrogen content. The linear equations are shown in Table III.

The elongation and reduction in area of the tensile specimens remained constant with varying nitrogen content.

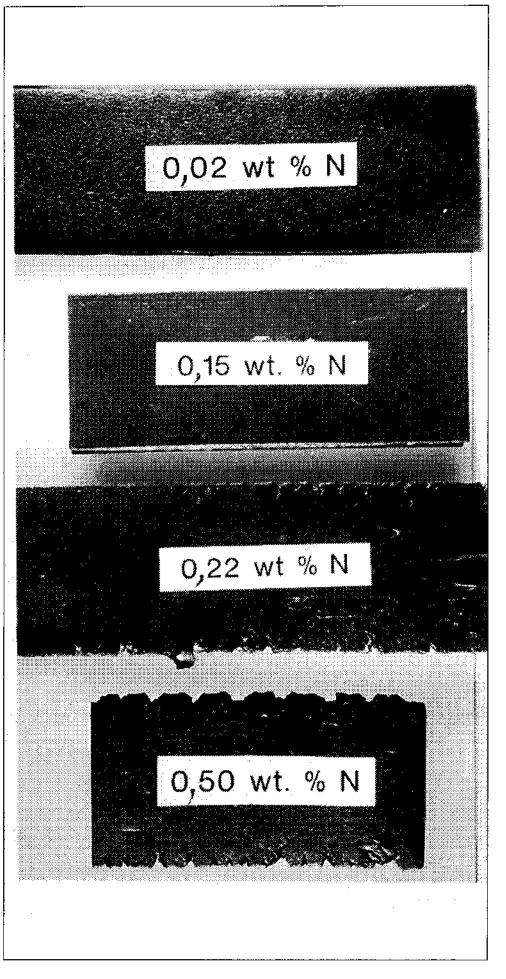

Figure 1—Appearance of the edges of hot-rolled samples with different nitrogen contents

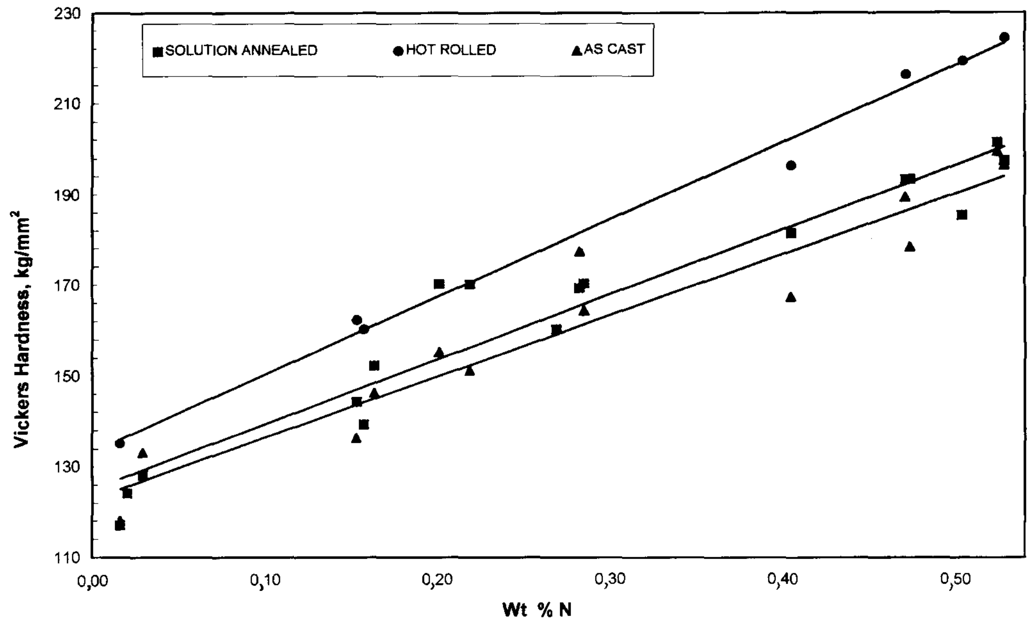

Figure 2—The influence of nitrogen on the hardness of AISI 310S

Table II

The results of regression analyses on the hardness-test results for material in three different conditions

Condition	Vickers hardness	Correlation coefficient
As-cast	HV_{10} = 121,3 + 141,2 (wt % N)	0,96
Hot-rolled	HV_{10} = 133,1 + 171,5 (wt % N)	0,99
Annealed	HV_{10} = 127,2 + 140,2 (wt % N)	0,94

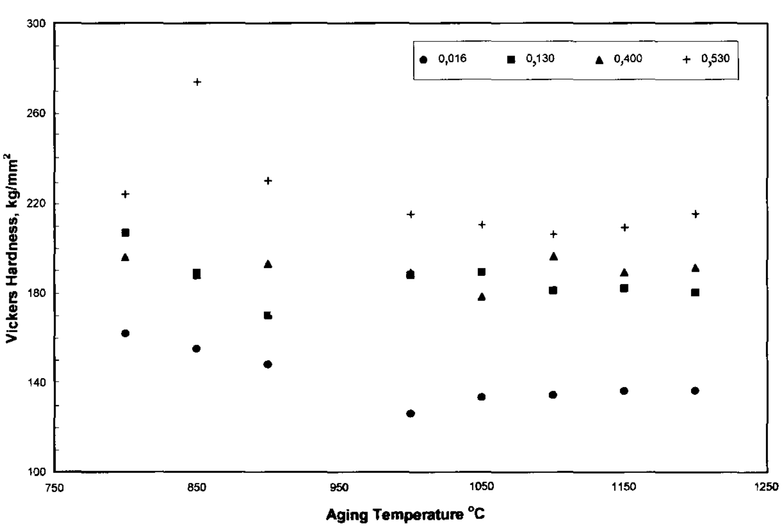

Figure 3—Variation of hardness with aging temperature

Impact properties

Charpy-impact values are shown in Figure 5. The results show considerable scatter, but a general trend of increasing impact value with increasing nitrogen content is discernible. In a consideration of Figure 5, it should be borne in mind that these values were obtained on subsize specimens with a cross-sectional area of 50 mm². Thus, it is evident that extremely tough material was obtained for nitrogen contents of up to 0,525 per cent by weight.

Fatigue properties

The results of the fatigue tests are presented in Figure 6. The rate of crack growth at a specific ΔK decreased with increasing nitrogen content. Regression analyses were carried out on the results to develop equations for the growth rate of stage II cracks (Table IV).

The power equations in Table IV demonstrate that the slopes of the curves decreased with increasing nitrogen content. Figure 7 shows that, at higher ΔK values, the rate of crack propagation decreased more rapidly with increasing nitrogen content. The values of da/dN decreased from about 30 x 10^{-5} to about 1 x 10^{-5} mm·c^{-1} for a value of ΔK equal to 25 MPa $\sqrt{m}$ and an increase in nitrogen content from 0,020 to 0,50 per cent by weight. However, the decrease for a ΔK of 35 MPa $\sqrt{m}$ is from about 16 x 20^{-5} to 2 x 10^{-5} mm·c^{-1} for the same increase in nitrogen content.

References

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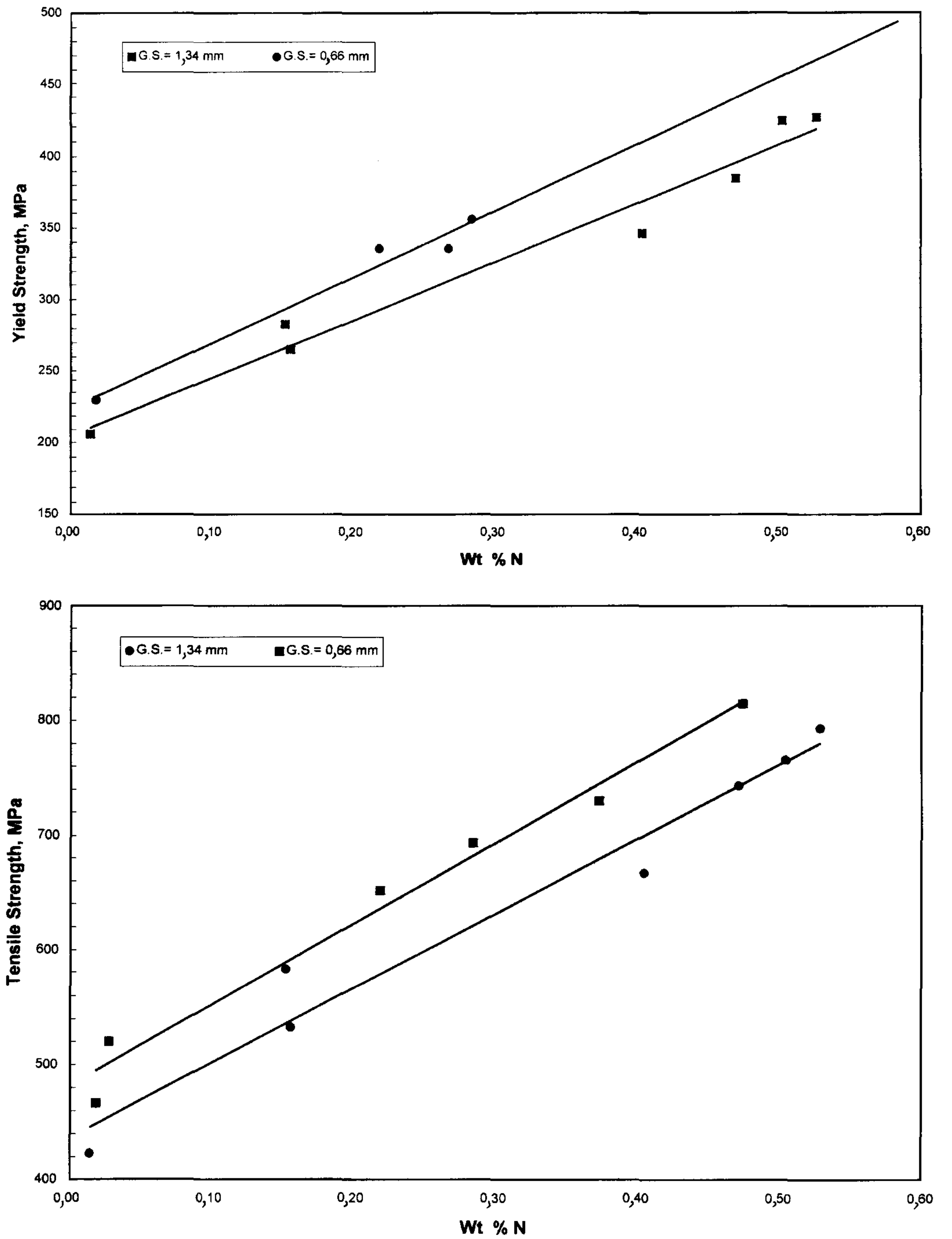

Figure 4—Effect of nitrogen on the yield and tensile strengths of AISI 310S of two different grain sizes

Precipitation characteristics

The precipitation characteristics of the materials were examined after they had been heat-treated at temperatures between 800 and 1200°C for 4 hours at intervals of 50°C. Material with a nitrogen content of less than 0,525 per cent by weight showed a coarsening of nitride particles. Material with a nitrogen content of 0,525 per cent exhibited precipitation of the lamellar (pearlite-like) constituent. This constituent appeared initially at the grain boundaries and spread through the matrix with increasing temperature up to 1050°C. At temperatures above 1050°C, this constituent re-dissolved in the matrix. Figure 8 shows material treated at different temperatures. The volume fraction of precipitate increased with temperature (Figure 8), as well as with time (Figure 9). The micro-indentation hardness of the pearlite-like constituent was 288 HV 0,2, while that of the matrix was 231 HV 0,2.

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Table III

Results of regression analyses on the tensile data

Grain size, mm	Equation	Correlation coefficient
Yield strength		
1,34	$\sigma_{Y.S.} = 203,9 + 408,4$ (wt % N)	0,99
0,66	$\sigma_{Y.S.} = 222,6 + 467,6$ (wt % N)	0,99
Tensile strength		
1,34	$\sigma_{T.S.} = 437,2 + 641,3$ (wt % N)	0,98
0,66	$\sigma_{T.S.} = 480,2 + 712,2$ (wt % N)	0,99

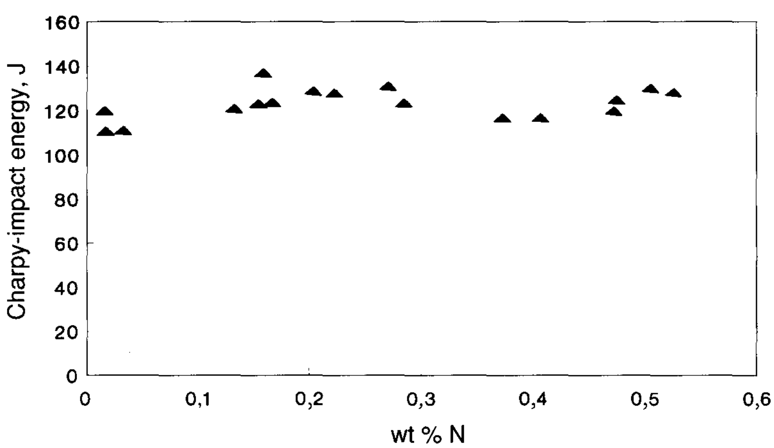

Figure 5—Effect of nitrogen content on the Charpy-impact energy of subsize specimens of 310S

12. Byrnes, M.L.G., Grujicic, M., and Owen, W.S. Nitrogen strengthening of a stable austenitic stainless steel. *Acta Metallurgica.*, vol. 35. 1987. pp. 1853–1862.
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X-ray-diffraction analyses of the material aged at 900°C showed that the lamellar constituent consisted of alternating layers of austenite and chromium nitride (Cr_2N), as shown in Figure 10. It should be noted that the (220) line of austenite at about 75° 2θ was resolved into two distinct peaks, indicating austenite of two different lattice parameters. The resolved peaks are superimposed on the original diffractogram in Figure 11, which shows the relevant part of the diffractogram in detail. It can be seen that the peak for austenite, γ, occurs at a lower angle than that for γ_1 and γ_2. From the positions and *d*-values of these three peaks, it is evident that the original austenite, γ, contained more nitrogen than the matrix austenite after aging, γ_1, which in turn contained more nitrogen than γ_2. It is clear that two different austenites were present: the matrix austenite, and the austenite associated with the lamellar constituent.

Fractography

The fracture surfaces of the fatigue-test specimens showed typical striations that are a visual record of the position of the crack front as this propagated through the material. This type of fracture surface is typical of conditions prevailing during the growth of stage II cracks.

Discussion

Hot-working and thermal processing of ingots

It is well known that the hot-workability of AISI 310S is limited. Previous studies[8] have shown that high nitrogen contents have an adverse effect on hot-workability. This can be combated by increased manganese content. It has been reported[9] that a manganese content of 2 per cent in a steel containing 0,15 per cent nitrogen is sufficient to give good hot-working characteristics. In the steel used in this investigation, the manganese content was 1,6 per cent, which may explain the relatively good hot-workability of the material.

Hot-workability is influenced by numerous other factors, such as macrostructure, hot-rolling temperature, and rolling conditions. Torkhov *et al.*[10] established optimum hot-rolling conditions for high-nitrogen steel containing 24 Cr-16 Ni-5,5 Mn. They showed that successful breakdown of the ingots could be accomplished at temperatures between 1090 and 1150°C.

Since the annealing was carried out in air, the question arises as to whether de-nitrogenization took place during this process. It has been indicated that, for high-nitrogen alloyed Cr-Ni austenitic steel, no de-nitrogenization is expected during hot-rolling and annealing unless the steel surface is heavily oxidized. Kikuchi *et al.*[11] report that, during annealing in air, pick-up of nitrogen is favoured rather than de-nitrogenization.

Torkhov *et al.*[10] studied the optimum annealing conditions under which all the particles of precipitate could be dissolved in a 24 Cr-16 Ni-5,5 Mn-0,6 N steel. The particles were completely dissolved after 4 hours at 1230°C or 2 hours at 1240°C. These annealing conditions compare well with the conditions used in the present study. The longer annealing time required in the latter was due to the lower temperature used (1200°C).

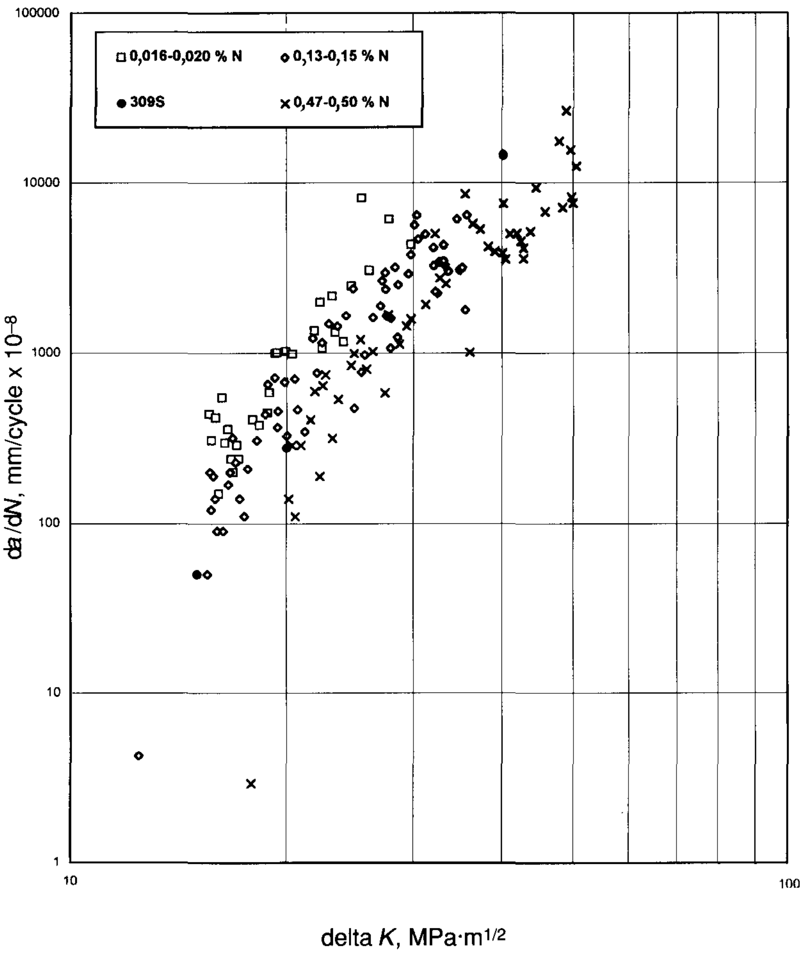


Figure 6—The effect of nitrogen on the fatigue properties of 310S (data for 309S are included for comparison)

Table IV

Results of regression analysis on the crack-growth data

Nitrogen wt %	Equation	Correlation coefficient
0,020	$da/dN = 1,89 \times 10^{-12} (\Delta K)^{5,12}$	0,92
0,13–0,15	$da/dN = 1,06 \times 10^{-11} (\Delta K)^{4,34}$	0,93
0,47–0,50	$da/dN = 1,51 \times 10^{-11} (\Delta K)^{4,07}$	0,93

Tensile properties

The equations in Table III show that nitrogen has a more pronounced effect on tensile strength than on yield strength. This is evident from the slopes of the lines in Figure 4. The difference, $(\Delta\sigma)$, between tensile and yield strength for a steel of specific grain size can be expressed as follows:

$$\Delta\sigma = (\sigma_{T.S.} - \sigma_{Y.S.}) = 257,6 + 244,4 \text{ (wt \%N)}.$$

This equation shows, in effect, that the strain-hardening characteristics of the material are strongly influenced by the nitrogen content, and that an increase in nitrogen results in more pronounced strain-hardening behaviour. Sandstrom[4] obtained similar results for steel containing 18 Cr–10 Ni.

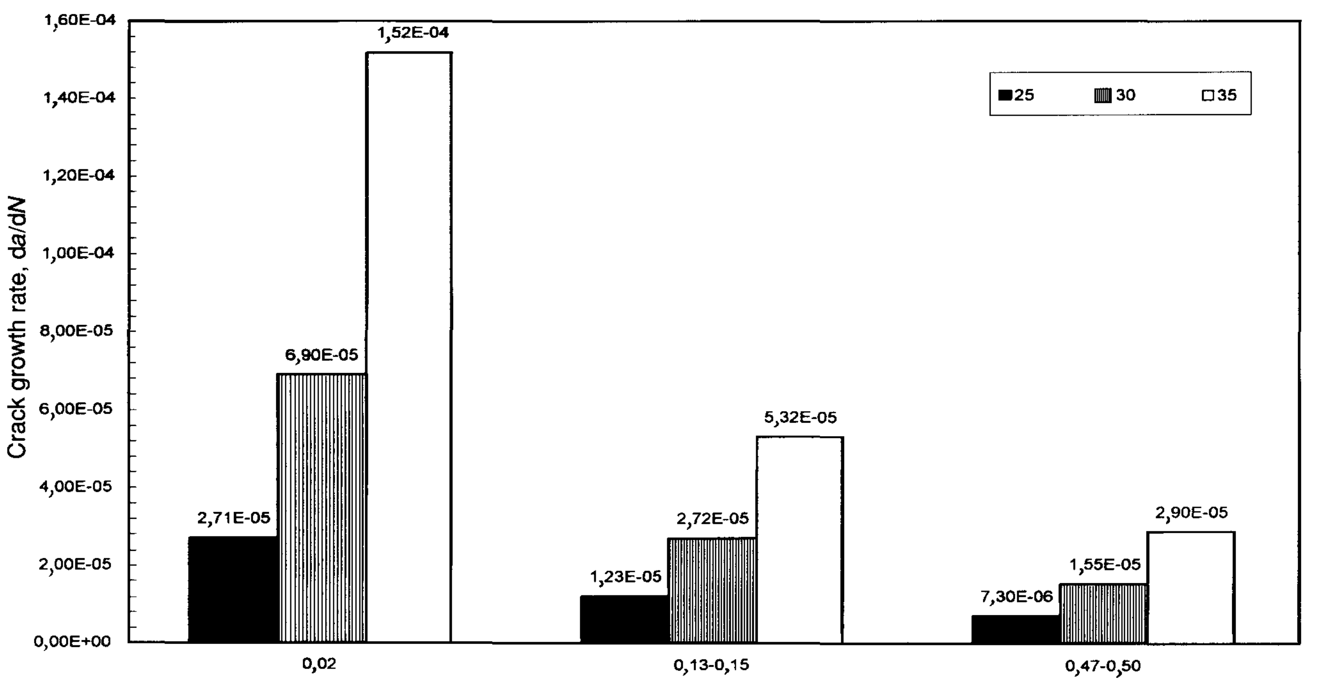


Figure 7—Variation in the rate of crack growth at different ΔK values with increasing nitrogen content

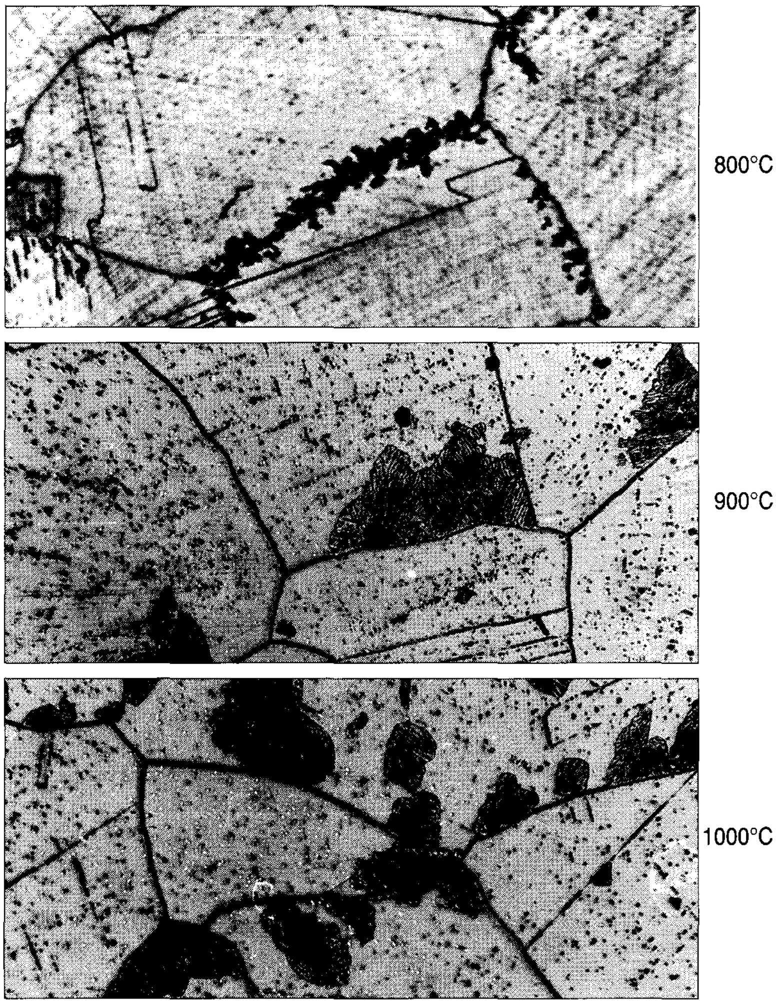


Figure 8—Lamellar type of precipitation at different aging temperatures x 100

The effect of grain size on strength is also clearly demonstrated in Figure 4. The lines for different grain sizes are not parallel, and the effect of nitrogen on strength is more pronounced in material of a finer grain size. This can also be demonstrated mathematically from the slopes of the equations, which increase with decreasing grain size. The increase in yield and tensile strength was respectively 408,4 and 641 MPa per percentage nitrogen by weight for a grain size of 1,34 mm. For samples with a grain size of 0,66 mm, the increase in the yield and tensile strengths was 467,6 and 712,0 MPa per percentage nitrogen by weight. The equations in Table III were used to generate values of yield strength for a range of nitrogen contents within the actual range of nitrogen contents obtained experimentally. The experimental data and the data generated as described were then used to give an equation relating yield strength to nitrogen content and grain size:

$$\sigma_{Y.S.} = 128,4 + 416,7\ (\text{wt }\%\text{N}) + 84,3\frac{1}{\sqrt{d}},$$

where d is in millimetres. The correlation coefficient (r^2) for this equation was 0,97, indicating a good fit.

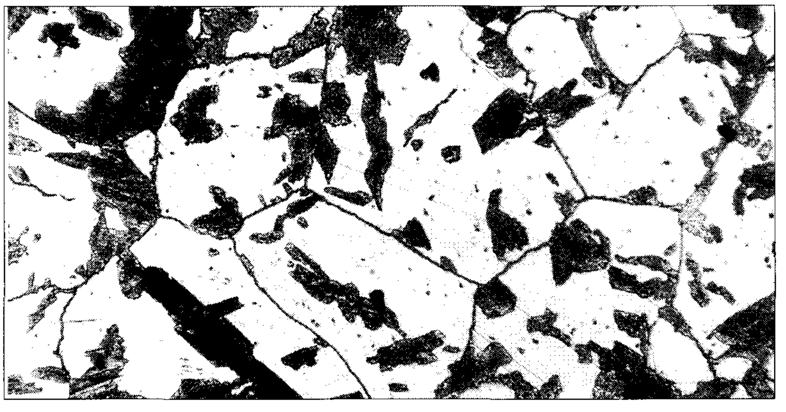
Previous studies[2] had indicated an increase in yield strength of 500 MPa per percentage nitrogen by weight. This value compares well with the value of 467,6 MPa determined in this study for material with a grain size of 0,66 mm. Figure 12 shows a comparison between the equation obtained in this study and those obtained by Pickering[3] and Norstrom[5]. Pickering's equation relates yield strength to a number of different elements (including nitrogen) in austenitic stainless steels, while Norstrom's equation was obtained from a study on steel AISI 316 only. Pickering and Norstrom worked on low-nitrogen steels compared with the steel used in the present study. Pickering's equation is linear, while Norstrom's follows a power law. It is clear from Figure 12 that there is good agreement between the results of this study and those of Pickering and Norstrom, even though the equations derived are somewhat different.

Three models have been formulated in relation to the effect of nitrogen on the strength of austenitic stainless steels: those based on the elastic interaction, the interaction between stacking faults and dislocations, and the short-range order.

The elastic-interaction model is based on the Fliecher theory, viz that solid-solution strengthening arises from the interaction of the strain field around point defects and the strain field of dislocations. The effect of changes on the shear modulus in the vicinity of point defects is also taken into consideration. Interstitial solid-solution strengthening results from the dilation of the lattice parameter and the variation of shear modulus with nitrogen content. The dilation factor, δ, is given as 0,20 in the literature[2,3,10,11] and appears to control the solid-solution strengthening effect of nitrogen.

The model based on the interaction between stacking faults and dislocations relies on the fact that nitrogen promotes the formation of planar arrays of dislocations, which is probably due to a decrease in the stacking-fault energy. This promotes interaction between partial dislocations and point defects, and results in higher yield strength. However, this interaction is not strong enough to entirely account for the strengthening obtained in nitrogen-containing steels[12].

4 h at 1000°C



10 h at 1000°C

Figure 9—The variation in volume fraction of lamellar precipitate with time at 1000°C (x 100)

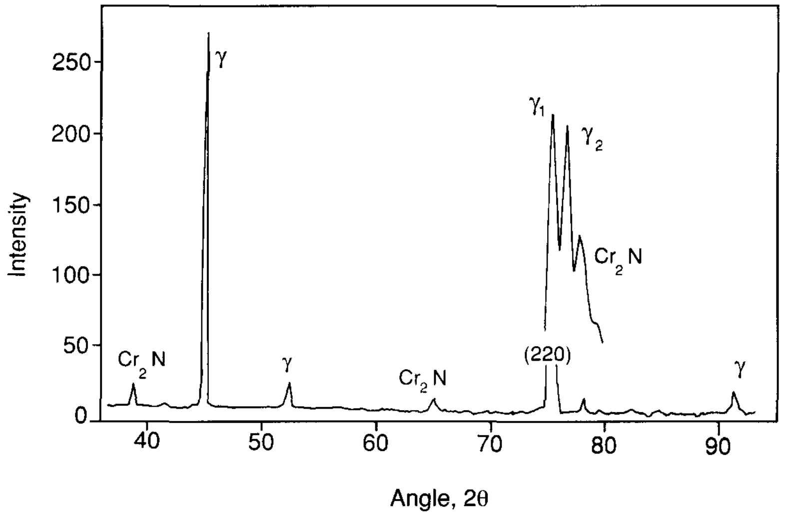


Figure 10—X-ray diffractogram of the sample with the cellular precipitate

The short-range order model is based on the results of neutron diffraction, which show that there is a possibility of short-range order among chromium and nitrogen atoms in austenitic stainless steels. If the stacking-fault energy does not change with the nitrogen content, the formation of planar arrays of dislocations can be explained by the short-range ordering of chromium and nitrogen atoms. This ordering could be due to a strong affinity between these atoms. However, at present, there is no proof of the existence of short-range ordering in these atoms.

Fatigue behaviour

According to the Paris law, the rate of crack growth is expressed as $da/dN = C(\Delta K)^n$. The parameter C is a function of the properties of the material. Table IV shows that C increased with increasing nitrogen content in the material used in this study. Generally, the fatigue properties of steels are related to their tensile strength.

However, in the case of steel AISI 309S, it was found[13] that the strength had no effect on the rates of crack growth. Since 309S and 310S steels are similar materials, changes in the strength of 310S might be expected not to have a significant effect on its fatigue properties. The changes in the rate of crack growth in 310S demonstrated in Figure 6 were not directly related to strength changes as a result of increasing nitrogen content. Therefore, the improvements in fatigue properties with increasing nitrogen content must have been due to changes in slip behaviour in the steel. This was probably due to the ability of nitrogen to promote the formation of planar arrays of dislocations, which impede cross-slip in the steel.

Conclusions

(1) The yield and tensile strength of the new alloys increased linearly with the nitrogen content. The yield strength was doubled by the addition of 0,48 per cent nitrogen by weight. The hardness of the new alloys increased linearly with increasing nitrogen content. The improvements in tensile properties appear to be due to elastic interactions and to interactions between stacking faults and dislocations in the lattice. Two empirical formulae were developed for the calculation of the yield and tensile strengths of the alloys as a function of nitrogen content.

(2) The tensile strength of nitrogen-containing 310S increased at a higher rate than the yield strength with increasing nitrogen content.

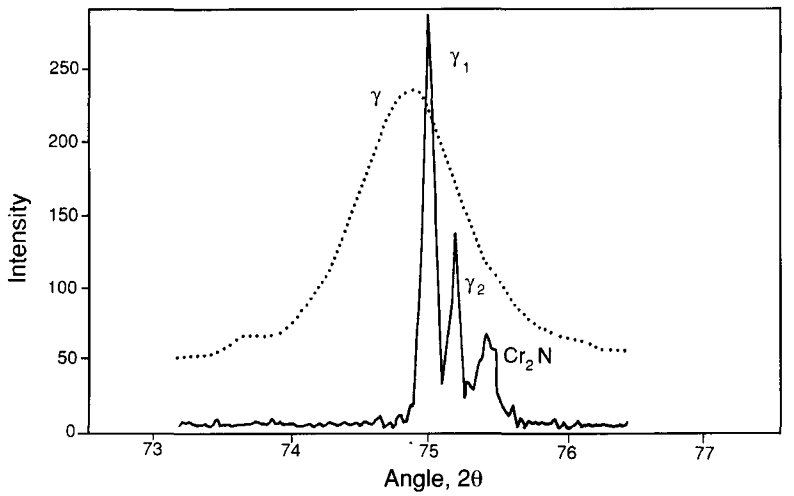


Figure 11—Comparison between the positions of the austenite (γ) peaks in a fully austenitic matrix with those of the austenitic matrix (γ_1) in material containing the lamellar constituent, and with those of the austenite in the lamellar regions (γ_2)

(3) The toughness of the material as measured by Charpy-impact tests was very high for nitrogen contents of up to 0,525 per cent by weight.

(4) The rate of fatigue-crack growth in the specimens decreased with increasing nitrogen content.

(5) A pearlite-like lamellar microstructure was observed in material with a nitrogen content of 0,525 per cent by weight after it had been aged at temperatures between 800 and 1050°C. The austenite associated with the lamellar precipitate had a lower nitrogen content than the matrix. ◆

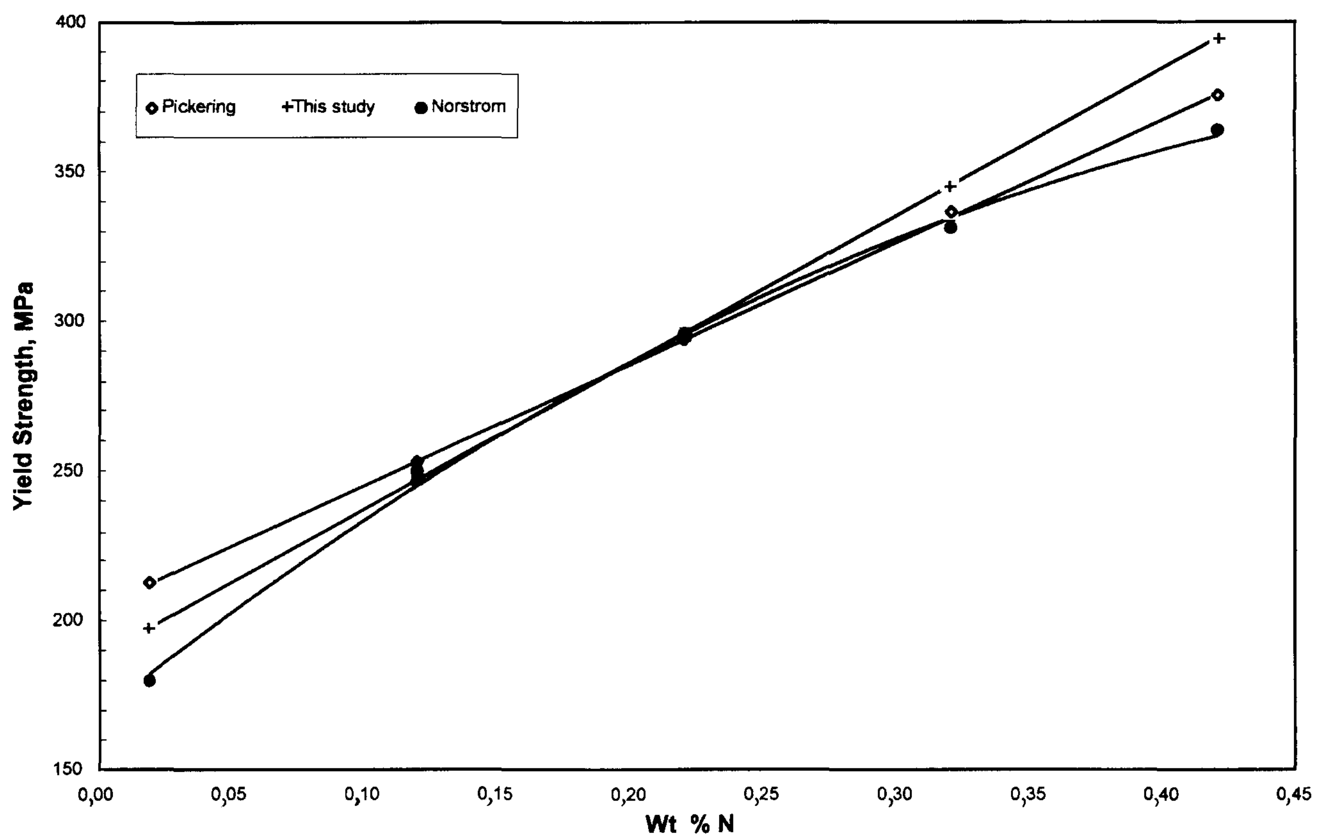


Figure 12—The variation in yield strength with nitrogen content, as shown by three different studies

Wits expertise paves the way for SA trade with Turkey*

Collaborative trade could be the result of a visit by Professor Neil James, head of physical metallurgy in the School of Process and Materials Engineering at the University of the Witwatersrand, to the Marmara Research Centre in Gebze, near Istanbul. Professor James recently accepted an invitation to visit the centre to participate in a NATO-funded research project dealing with the powder metallurgy of hard metals. The project aims to provide Turkey with the expertise to produce hard-metal rolls for steelmaking.

Together with world expert Professor Silvana Luyckx of the Schonland Research Centre, Professor James lectured and assisted in laboratory experiments on the processing of hard metals and mechanical testing for fatigue and fracture toughness.

He says South Africa's research expertise and sophisticated industry have made this country a sought-after centre of excellence in the field. He hopes to establish a collaborative project between the Marmara Research Centre and the School of Process and Materials Engineering at Wits University on tungsten carbide–cobalt hard metals. The ultimate objective, he adds, is for Turkey to import expertise, powders, and possibly products from South Africa.

In his capacity as director of the Fracture Research Group at Wits, Professor James has written the guest editorial for a special issue of the *International Journal of Fatigue*, which is to publish a selection of the papers presented at the 'Fracture '94' conference, that was held in Johannesburg in November last year. The conference was strongly inclined towards the transfer of technology to industry.

'A particularly pleasing aspect of Fracture '94 was the increase in the number of overseas visitors presenting papers', says Professor James in his editorial. He adds that the papers published in the special issue of the *International Journal of Fatigue* illustrate several potential problems in failure analysis, presenting the results of industrially funded case studies arising from failure analyses and highlighting some fundamental aspects of research into the growth of fatigue cracks. ◆

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Talks bode well for US collaboration with Wits University*

A visit by a presidential mission of the Office of Energy Research in the USA to the Faculty of Engineering and Department of Physics at the University of the Witwatersrand looks set to open up avenues of co-operation.

After a tour of the research facilities, Dr Martha Krebs, director of energy research at the US Department of Energy, said Wits was a very capable university and that there might well be opportunities for collaboration. 'The challenge for both of us is finding ways to extend these opportunties to disadvantaged students', she added, the talks with faculty representatives having revealed many research matches that could be followed up.

Talks also centred on the need for collaboration between historically white and historically black universities, and between universities and technikons, in South Africa. Here the USA could give advice based on experience.

The presidential mission on sustainable development was led by Secretary Hazel O'Leary. The Office of Energy Research of the US Department of Energy (DOE) had participated in the mission to seek opportunities for mutually beneficial scientific partnerships with the South African government, academia, national laboratories, and industry. The DOE spends between 2,5 billion and 3 billion dollars on research annually.

Dr Krebs pointed out that the visit of the presidential mission was of a preliminary nature, and would be followed at the end of this year by visits involving detailed discussions. ◆

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International recognition for Wits engineering degrees*

The London Institution of Mining and Metallurgy (IMM) has formally accredited the degrees in mining engineering and minerals processing awarded by the Faculty of Engineering at the University of the Witwatersrand. This means that an international institution has formally approved the degrees, giving these graduates recognition and acceptance in the workplace overseas. This also represents a testimony of the high quality of the courses.

The accreditation comes after a visit by IMM representatives from the UK to the Department of Mining Engineering and the Department of Metallurgy and Materials Engineering late last year. According to Professor Jan Reynders, dean of the Faculty of Engineering, it makes Wits the only university in South Africa at which all the engineering degree courses offered (9 in all) have gained formal international recognition. ◆

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