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A. Cigada,[1] *B. Mazza,*[1] *G. A. Mondora,*[1] *P. Pedeferri,*[1]
G. Re,[1] *and D. Sinigaglia*[1]

Localized Corrosion Susceptibility of Work-Hardened Stainless Steels in a Physiological Saline Solution

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ABSTRACT: This paper describes: (1) an electrochemical technique for crevice corrosion testing and (2) the results of a study concerning the susceptibility to crevice and pitting corrosion of work-hardened AISI 316L and 304L stainless steels in a sodium chloride buffered solution simulating the aggressiveness of the human body. In particular, the importance of the deformation degree and the orientation of the specimen surface under study with respect to the deformation direction is shown and discussed in relation to characteristic structural aspects.

KEY WORDS: implant materials, corrosion, crevice corrosion, pitting, corrosion tests, stainless steels, cold plastic deformation

Within the framework of a systematic study of the influence of cold plastic deformation on susceptibility to localized corrosion of austenitic stainless steels, pitting and crevice corrosion in aggressive media of different compositions have been investigated.

As regards the topic of this publication, it is well known that crevice corrosion is considered to be the main cause of failure in the application of stainless steel orthopedic implants. This form of attack occurs at interface contacts, for instance, between the screws used for implant fixation and their seats. AISI 316L stainless steel is particularly susceptible to crevice corrosion: according to Greene [*1*],[2] up to 91 percent of multicomponent

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[1] Assistant professor, associate professor, assistant professor, associate professor, assistant professor, and associate professor, respectively, Istituto di Chimica-fisica, Elettrochimica e Metallurgia del Politecnico di Milano, Milan, Italy.

[2] The italic numbers in brackets refer to the list of references appended to this paper.

devices made with this type of steel would be so affected. Still more recently, Levine and Staehle [*2*] state that "of all metal used in multicomponent devices, Type 316L stainless steel is the most susceptible to crevice attack."

As far as pitting is concerned, even stainless steels containing molybdenum frequently can be susceptible, under certain conditions. Susceptibility to this form of corrosion occurs because of other factors. One example of such factors is material work-hardening, which lowers the critical pitting potential.

Several observations have been reported on the unfavorable effects of the cold working on the resistance to localized corrosion of AISI 316L steel in the human body or in artificial solutions simulating body fluid aggressiveness, but only recently have systematic studies been carried out [*3*].

This paper describes the results of an *in vitro* study concerning the susceptibility to crevice corrosion of work-hardened AISI 316L steel. A comparison is made with the results of previous analogous pitting susceptibility studies [*4–6*]. Corrosion behavior is correlated with the structure of work-hardened steels. For the sake of comparison, AISI 304L steel also has been studied.

Experimental

Materials

Table 1 lists the composition of the stainless steels studied. The materials were first annealed at 1050°C for 1 h and water quenched (solution heat-treatment), then subjected to cold plastic deformation by either tension or rolling. Deformation by tension is more significant from a theoretical point of view (for example, the situation is better defined concerning the distribution of stresses in the course of the deformation process [*7*]), whereas deformation by rolling is more significant from a practical point of view.

The main characteristics of the deformation processes are listed in Table 2 together with the deformation degrees obtained (expressed in terms of reductions in the cross-sectional areas of the pieces). A general idea of the material work-hardening effects is given by the results of the Vickers hardness tests (Fig. 1).

Cold working at liquid nitrogen temperature (−196°C) improves the mechanical strength of the steels under study to a very high degree, but the corresponding influence on the localized corrosion resistance is insufficiently known. Moreover α'-martensite in large quantities is obtained under these deformation conditions (Fig. 2), and effects of this phase can be identified.

TABLE 1—*Chemical compositions of the stainless steels studied.*

Steel Type	Type of Cold Working	Composition, %								
		C	Si	Mn	P	S	Cr	Ni	Mo	N
AISI 316L	tension	0.022	0.43	1.51	0.033	0.023	16.80	10.65	2.90	0.033
	rolling	0.026	0.41	1.24	0.008	0.011	16.10	10.90	2.20	0.034
AISI 304L	tension	0.025	0.45	1.39	0.023	0.021	18.60	8.75	0.50	0.036
	rolling	0.020	0.41	1.40	0.032	0.013	18.10	10.30	0.32	0.039

TABLE 2—*Main characteristics of the deformation processes and the deformation degrees obtained.*

Deformation			
Type	Temperature, °C	Rate[a]	Degree,[b] %
Tension	25	0.05 kg/mm^2 s (rate of increase in the applied stress)	10 15 30
	−196	0.13 kg/mm^2 s (rate of increase in the applied stress)	9 13 19
Rolling	25	4 m/min (speed of the unloaded rolls)	10 30 50
	−196	4 m/min (speed of the unloaded rolls)	10 30 50

[a] As indicated in the different cases.
[b] Reduction in the cross-sectional area.

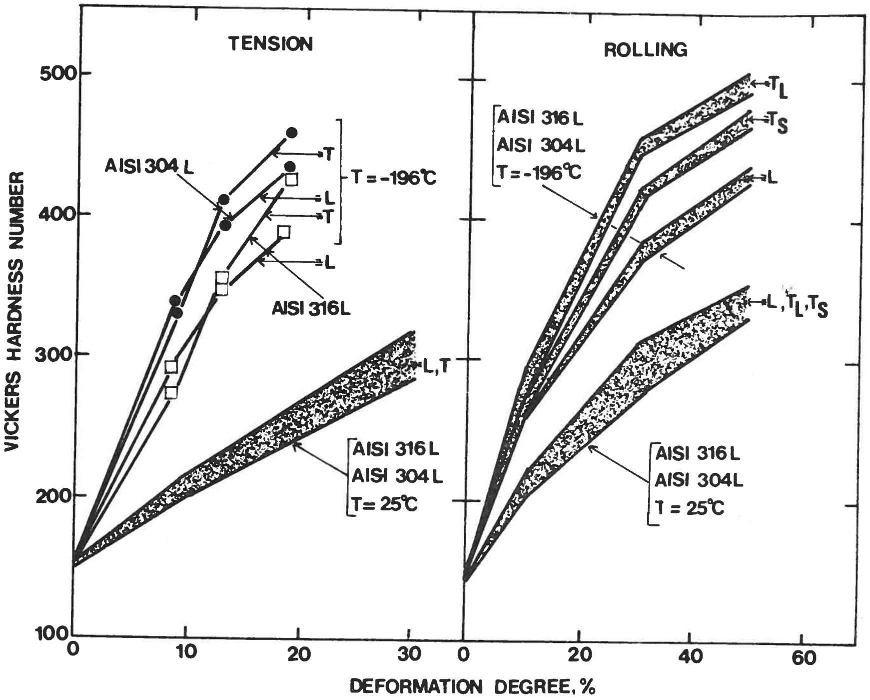

FIG. 1—*Vickers hardness number (VHN) for AISI 316L and 304L steels as a function of the deformation degree for different types and temperatures of cold working and for different orientations of the specimen surface to the deformation direction; L = longitudinal, T = transversal, T_S = short-transversal, T_L = long-transversal surface.*

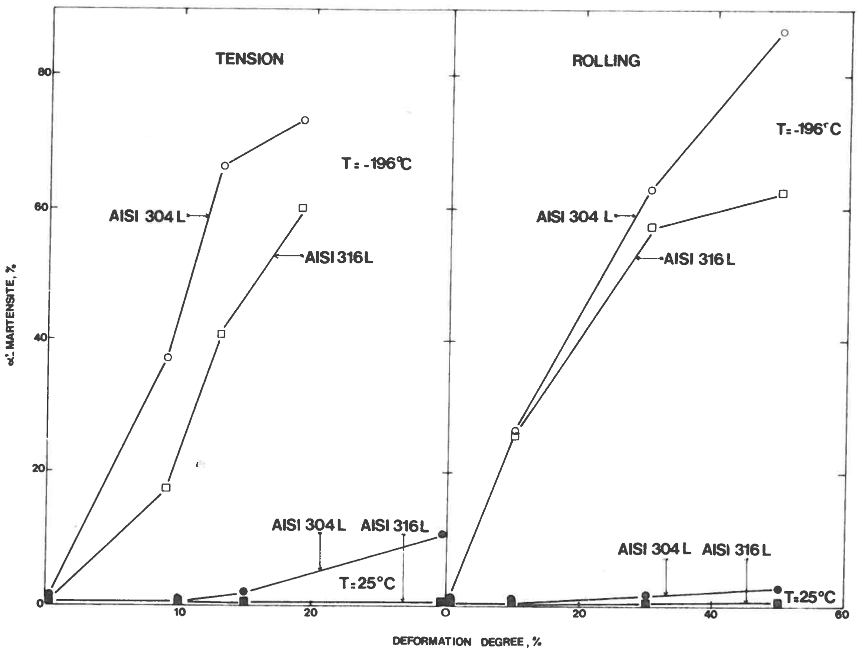

FIG. 2—*α'-martensite percentage (determined by magnetic measurements* [8]) *in AISI 316L and 304L steels as a function of the deformation degree for different types and temperatures of cold working.*

Specimens Preparation

Specimens were cut to obtain different surface orientations with respect to the deformation direction. Longitudinal (L) and transversal (T) surfaces in the case of deformation by tension, and longitudinal (L), long-transversal (T_L) and short-transversal (T_S) surfaces in the case of rolling, were considered.

Surface preparation of the specimens was accomplished by the following operations:

1. Wet grinding with emery papers to a 800-mesh finish.
2. Electropolishing in a solution of ethanol and perchloric acid.
3. Polishing with diamond pastes with decreasing grain size down to 1 μm.
4. Rinsing in denatured alcohol and then in distilled water, in the presence of ultrasonic vibrations.

Specimens were then stored in a desiccator for at least 12 h.

Test Environment

The tests were conducted in an artificial "physiological" solution, and the solution composition was as follows: sodium chloride ($NaCl$), 8.74 g/litre; sodium bicarbonate ($NaHCO_3$), 0.35 g/litre; dibasic sodium phosphate (Na_2HPO_4), 0.06 g/litre; monobasic sodium phosphate (NaH_2PO_4), 0.06 g/litre. This solution was buffered at a pH of 7, and its temperature was maintained at 38°C. Nitrogen was bubbled continuously to remove the air.

Test Cell and Electrochemical Procedure

The electrochemical cell and electrode assembly used for crevice corrosion testing are shown in Fig. 3. A reproducible crevice was produced by pressing a shielding probe ending in a spherical head (approximately 4 mm in diameter) against the surface of the working electrode. The potential within the crevice could be measured by means of a capillary through the spherical head, whereas the open surface of the working electrode was connected to the potentiostat through a capillary probe of the Luggin-Haber type with its tip positioned at a distance of a few millimetres. Reference electrodes of the type Hg, Hg_2SO_4/K_2SO_4 (saturated) [saturated sulfate electrode (SSE)], were used. The potential of the SSE versus a standard hydrogen electrode is +642 mV at 25°C.

Crevice corrosion susceptibility was evaluated by means of the following procedure. In order to obtain reproducibile passivity, the working electrode was first polarized at −600 mV versus SSE for 20 min, the shielding probe being lifted up. The latter was then pressed against the surface of the working electrode, whose potential (E_{open}) was then scanned upward (in the more noble direction) at a rate of 120 mV/h until crevice corrosion was indicated. Then potential scanning was stopped and crevice corrosion allowed to propagate to a certain extent potentiostatically. Polarization current density (i) and potential within the crevice ($E_{crevice}$) were recorded continuously versus time: their behavior is shown in Fig. 4.

In the absence of crevice corrosion phenomena on the electrode with the applied crevice, the polarization current density is negligible (lower than 1 $\mu A/cm^2$, in accordance with the passive state, under the working conditions of our experiments, of the stainless steels studied) and the potential within the crevice coincides with the potential of the open surface of the electrode.

The onset of crevice corrosion is indicated by a noticeable polarization current rise (over 10 $\mu A/cm^2$) but especially by a corresponding drop in the potential within the crevice, which, at this point, becomes different from the outside potential controlled by the potentiostat. In this way it is possible to determine the potential at which crevice corrosion, under the shielding

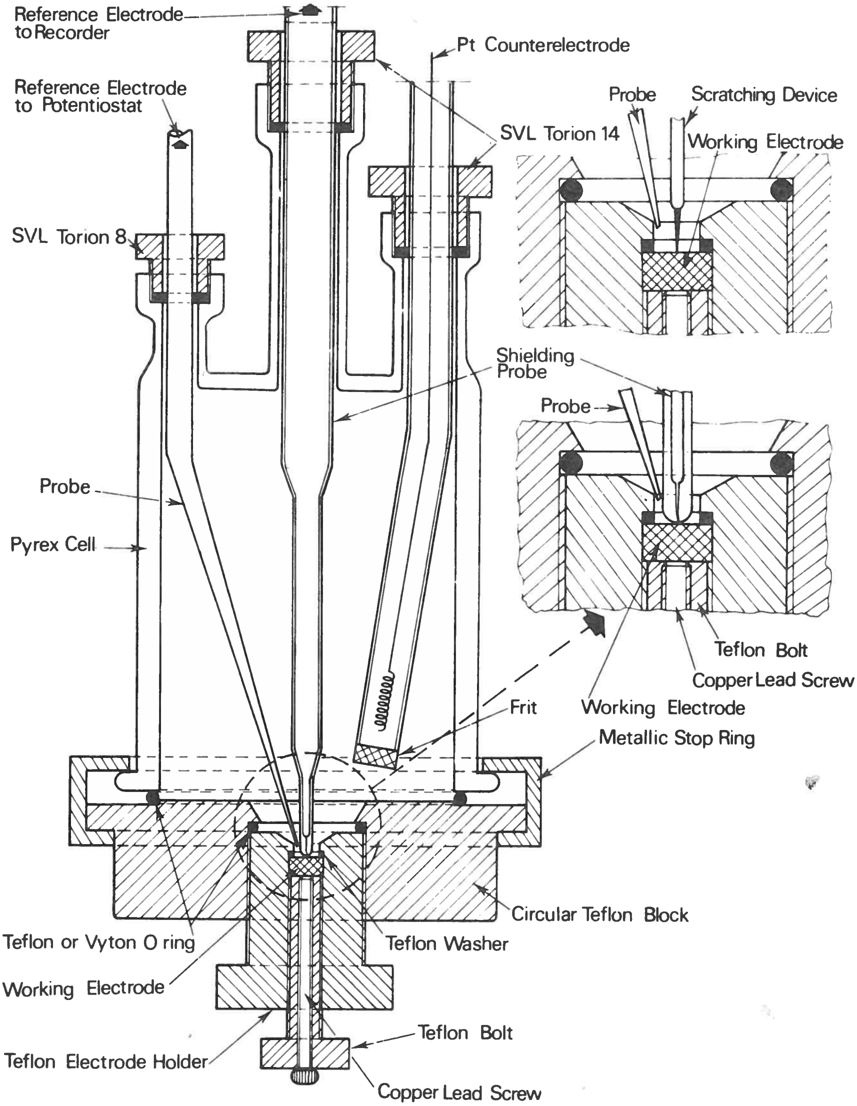

FIG. 3—*Electrochemical cell and electrode assembly (cross sections) used for crevice corrosion testing. Also the scratching device used to evaluate the pitting susceptibility* [4-6] *is shown.*

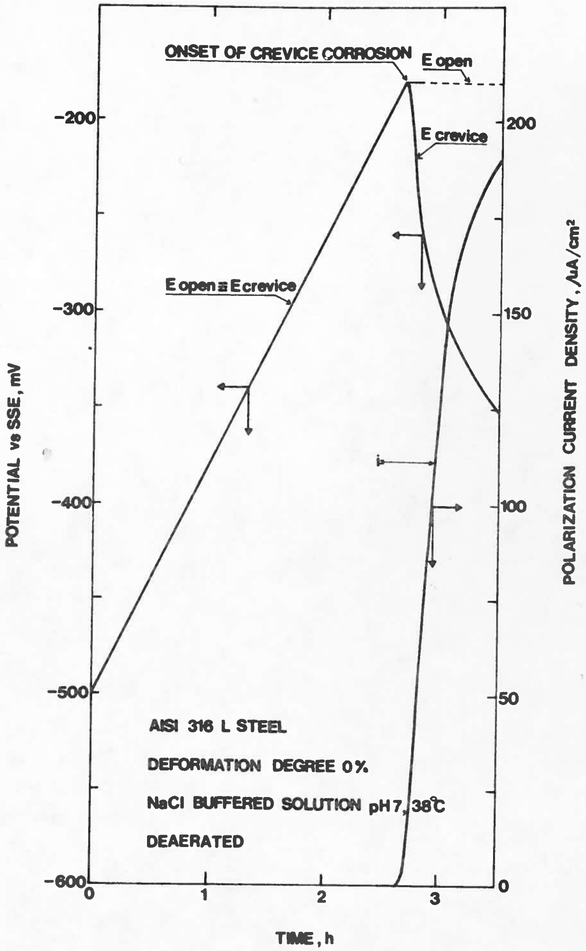

FIG. 4—*Circulating current* (i) *and potential within the crevice* ($E_{crevice}$) *as a function of time during potentiodynamic anodic polarization of a passive electrode with an applied crevice. The upward scanning of the outside potential* (E_{open}) *is stopped at the onset of crevice corrosion (within a few mV). Potential values are referred to the saturated mercury sulfate electrode (SSE), whose potential with respect to the standard hydrogen electrode is +642 mV at 25°C.*

probe, initiates independently the onset both of crevice corrosion under the Teflon washer, which defines the exposed electrode area, and of small repassivating pits on the open surface of the electrode. These last two circumstances have a small effect on the previously defined critical crevice corrosion potential only if the associated circulating current is small, that is, less than about 10 $\mu A/cm^2$.

The electrode potential values at which crevice corrosion occurs were reproducible within the limits of 50 mV at the most.

By keeping all parameters (including the geometrical ones) and test conditions constant, the procedure just described allows establishing a relative scale of susceptibility to crevice corrosion for a series of materials which differ from one another in chemical composition or structure, and the results obtained should be interpreted only in these terms [*9*].

After the electrochemical tests, the specimens were removed from the assembly and inspected for corrosion damage. The tests were considered valid only if a deep crevice attack was present under the shielding probe, whereas crevice corrosion under the Teflon washer or pitting corrosion were negligible. No observation could be made on nucleation sites.

Results and Discussion

Potentials at which crevice corrosion occurs under the considered conditions are shown in Figs. 5–7. For the sake of comparison the critical pitting potentials, determined in previous research work [*4–6*] by means of the Pessall and Liu [*10*] "scratching" technique,[3] are plotted.

By increasing the degree of deformation, resistance to crevice corrosion generally gets worse, more especially for the steels deformed at the liquid nitrogen temperature. However, in the latter case a recovery in resistance to crevice corrosion may be shown at the highest degrees of deformation.

An anisotropic behavior of specimen surfaces with different orientations to the deformation direction should be emphasized: the surfaces parallel to the deformation direction always exhibit a higher resistance than the transversal ones (in particular for the cold-rolled AISI 316L steel, the relative order of resistance is $L > T_S \geq T_L$). This anisotropy is particularly marked for the steels deformed at room temperature and increases with the degree of deformation.

It is well known that the susceptibility to crevice corrosion is lower (that is, the potential at which crevice corrosion occurs is more noble) for the 316L steel than for the 304L one, both in the undeformed state and in the work-hardened state of the material.

As regards the comparison between the results of the pitting and crevice corrosion tests, it may be pointed out that the corresponding curves of the "critical" potentials versus deformation degree are qualitatively quite similar (with the exception of the previously mentioned possible presence of a minimum in the crevice corrosion curves). From a quantitative point of view, the critical potentials for the onset of crevice attack are always less noble than the critical pitting potentials, which confirms the greater practical importance of crevice corrosion as a cause of failure in implant devices.

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[3] That is, the potentials above which a scratch in the surface film fails to repassivate.

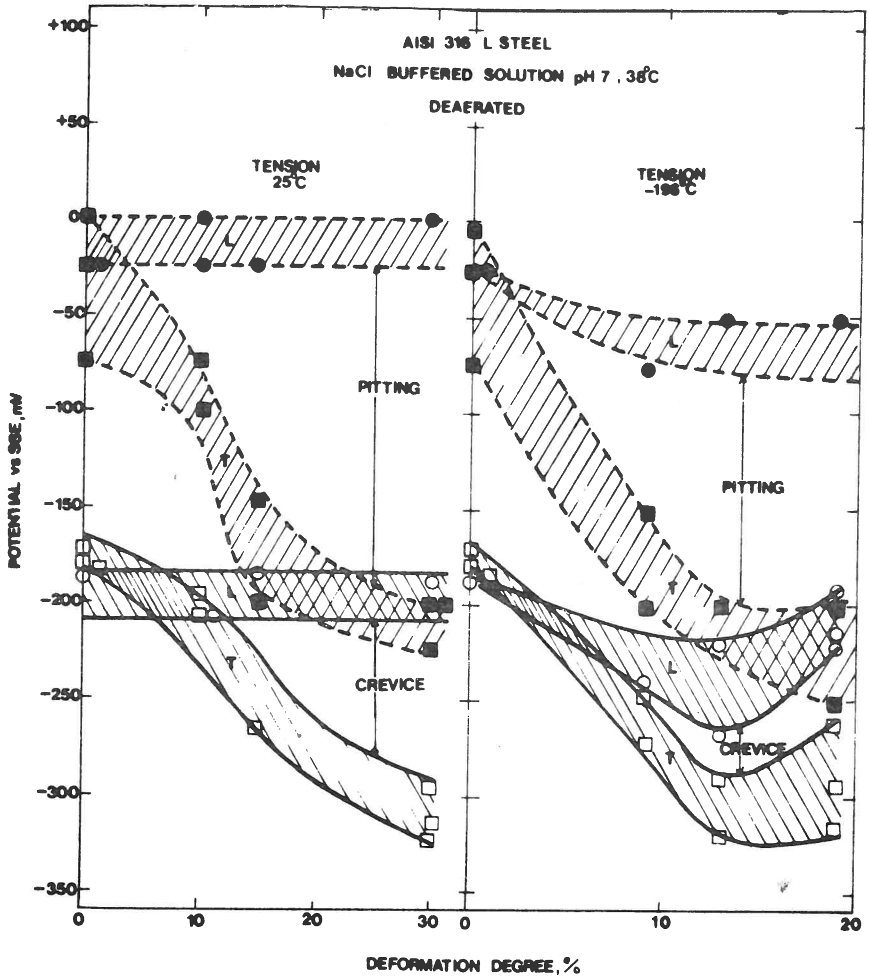

FIG. 5—*Potential at which crevice corrosion occurs as a function of the deformation degree for different orientations of the specimen surface to the deformation direction, in the case of AISI 316L steel deformed by tension at different temperatures* (○ *longitudinal,* ☐ *transversal surface). For the sake of comparison, also critical pitting potential* [4-6] *is plotted* (● *longitudinal,* ■ *transversal surface).*

As a whole, these results appear also to confirm the more general statement [*11*] that stainless steels which are immune to crevice corrosion are also immune to pitting corrosion at the same potential, chloride concentration, and temperature.

An interpretative scheme of the previously described results should consider the different passivation conditions of the specimens used for pitting

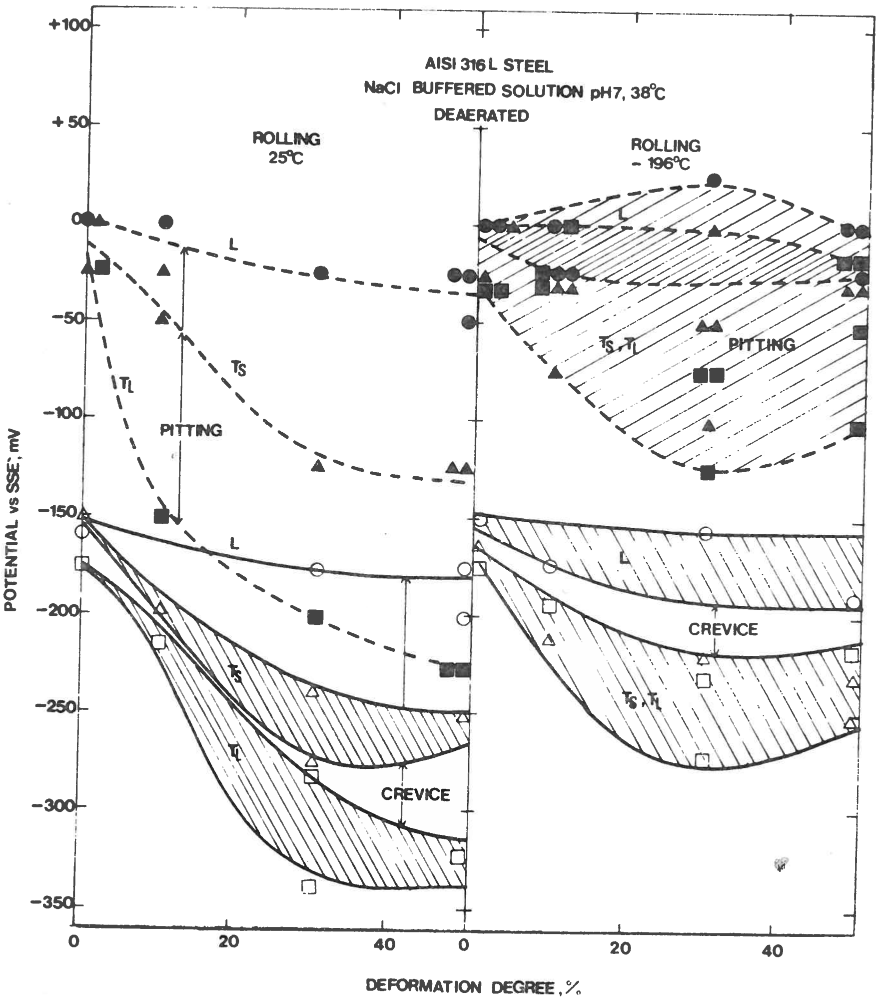

FIG. 6—*Potential at which crevice corrosion occurs as a function of the deformation degree for different orientations of the specimen surface to the deformation direction, in the case of AISI 316L steel deformed by rolling at different temperatures* (○ *longitudinal,* Δ *short-transversal,* □ *long-transversal surface). For the sake of comparison, also critical pitting potential* [4–6] *is plotted* (● *longitudinal,* ▲ *short-transversal,* ■ *long-transversal surface).*

or crevice corrosion testing: in the former case a passivation pretreatment in a solution of nitric acid (concentration, 30 percent; temperature, 55°C; duration, 30 min) was carried out [*4–6*], while in the latter case, the specimens were passivated potentiostatically in the same test solution. As a matter of fact, for the specimens prepassivated in a solution of nitric acid no attack under the applied crevice occurred in the given test environment within a testing period compatible with the minimum scanning rate of the

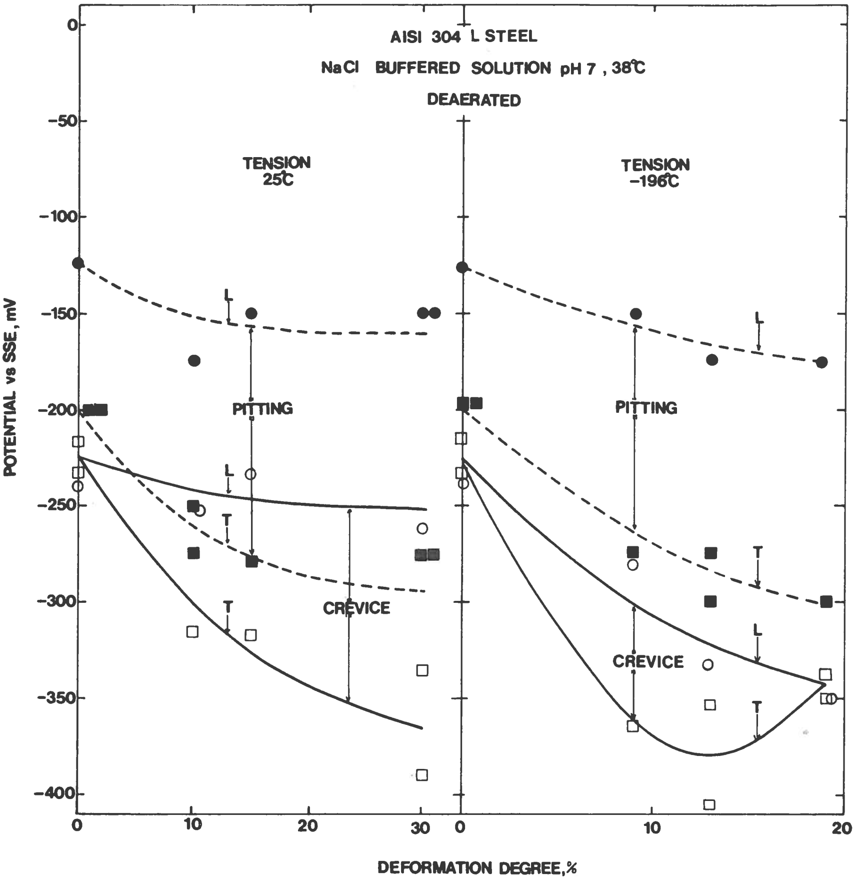

FIG. 7—*Potential at which crevice corrosion occurs as a function of the deformation degree for different orientations of the specimen surface to the deformation direction, in the case of AISI 304L steel deformed by tension at different temperatures* (○ *longitudinal,* ☐ *transversal surface). For the sake of comparison, also critical pitting potential* [4-6] *is plotted* (● *longitudinal,* ■ *transversal surface).*

potentiostat, unless an accidental moving of the shielding probe caused a scratch in the surface passivating film: also in this case of "scratching in the crevice" the electrode potential above which the scratch failed to repassivate was always less noble than the corresponding potential for "free scratching."

In any case, even independently of any mechanical damaging of the film, it would appear indisputable that the aptitude to local repassivation of discontinuities in the protective surface film of these steels is lowered due to the presence of an applied crevice, that is, an applied crevice aids the formation of pits. Thus the hypothesis that crevice attack nucleates from pitting initiation sites inside the shielded area can be shared by us as far as our experimental conditions and results are concerned.

As regards the relationship between the previously described aspects of the localized corrosion behavior of the steels considered and the structural effects of the cold plastic deformation, the presence of α'-martensite (even if it becomes far more prevalent, as shown in Fig. 2 for the cold working at liquid nitrogen temperature) has very little importance. Thus other structural aspects different from dislocations, deformation bands and phase transitions must be considered.

As a matter of fact, the great role played by the nonmetallic inclusions, in particular the sulfide inclusions, in the susceptibility to pitting of stainless steels is widely acknowledged [*12*]. According to this point of view, the sulfide dissolution results in microcavities from which the pitting propagation can occur.

It is also established that during plastic deformation microvoids can occur in the steel around the inclusions [*13,14*]. Moreover the inclusions themselves (which in the as-cast state are spherical and randomly distributed) deform to give triaxial ellipsoids with the major axis on the longitudinal sections and with the minor axis on the transversal ones [*15*]. As a result, the transversal sections show a higher density of inclusions and a shape of inclusions more favorable to pitting nucleation and propagation than the longitudinal sections [*16*]. This would account for the previously described anisotropic effects on the localized corrosion behavior of the deformed steels. Also the anisotropic effect sometimes shown by the materials undeformed by cold working (see, for example, the results of the pitting tests [*4*]), can be thus explained by the influence of operations undergone independently from cold working (for example, hot-rolling), an influence that is not cancelled by the solution heat-treatment.

As an interpretative conclusion to our results we could agree with those researchers which suggest that pitting and crevice corrosion are analogous processes but on different scales. Pitting can be considered as a microcrevice which propagates from the microcavities left by the dissolution of sulfide inclusions. On the other hand, crevice corrosion at macrosites nucleates from these microcrevices inside the shielded areas.

From a practical point of view we could say that a more accurate control of the production process, especially as regards the content of nonmetallic inclusions, could lead to improved resistance to localized corrosion of the stainless steels considered in the given environment. This corresponds to what other researchers state [*17*], that is, that the new extra-low interstitial (ELI) high-chromium (plus molybdenum) ferritic stainless steels, such as the Types 29-4, 26-1, and 21-3, appear to be quite promising as materials for implant surgery. As a matter of fact the control of interstitial elements, such as nitrogen, carbon, and hydrogen, also improves the quality of the steel as regards nonmetallic inclusions.

Conclusions

Resistance to crevice corrosion of work-hardened AISI 316L and 304L steels in a sodium chloride buffered solution simulating the aggressiveness of the human body has been tested by means of an electrochemical technique. The main results of this investigation can be summarized as follows:

1. Cold working generally lowers resistance to crevice corrosion.
2. Specimen surfaces parallel to the deformation direction always exhibit a higher resistance to crevice corrosion than the transversal ones (anisotropic effect).
3. These crevice corrosion results are qualitatively quite similar to the ones concerning the resistance of the steels studied for pitting corrosion in the same solution.
4. The presence of a crevice lowers critical potentials for the onset of localized attack.

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DISCUSSION

B. C. Syrett[1] (*written discussion*)—I think the authors are to be congratulated on a fine piece of work. I was particularly interested in the observed differences between the critical potentials for pitting and crevice corrosion. Since the mechanics of pit propagation and crevice corrosion propagation are essentially identical, we must look for some difference in the initiation process. Just prior to the onset of crevice corrosion, you indicate that the potential within the crevice is equal to the potential of the exposed surface. Thus increases in the chloride content of the crevice electrolyte, or decreases in the pH or dissolved oxygen appear unlikely. Do you have any explanations for the difference in the critical potentials for pitting and crevice corrosion? Could the difference be a result of the different surface pretreatments given to the pitting and crevice corrosion specimens (a nitric acid prepassivation for the pitting treatment specimens and a potential static passivation in the test solution for the crevice corrosion specimens). Certainly work in our laboratory indicated that the susceptibility to initiation was reduced greatly when Type 316L stainless steel was given a nitric acid prepassivation treatment. Secondly, contrary to your belief, I would like to suggest that the presence of α'-martensite sometimes has an important effect on crevice corrosion and pitting resistance.

While I agree that other effects of cold work were overriding at low levels of deformation, it would appear that at higher levels, the presence of martensite is often beneficial (for example, compare the 25°C and −196°C curves for the cold-rolled Type 316L stainless steel). A similar effect was demonstrated in our laboratory when we cold worked several TRIP steels and measured the change in the critical pitting potentials. TRIP steels are metastable, austenitic, stainless steels that readily transform partially to martensite on cold working at 25°C. We found that the critical pitting potential of each TRIP steel was increased considerably with cold work, indicating that the detrimental effects of cold work were more than compensated for by the production of the martensite.

A. Cigada et al (*authors' closure*)—Certainly the different surface pretreatments influence nucleation and propagation of localized corrosion, but such a difference could be evidenced only by operating always in the same

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[1] SRI International, Menlo Park, Calif. 94025.

way. On the contrary, in our work we compared two different parameters (that is, crevice corrosion and pitting critical potentials) measured in different ways.

We think that the difference between the two critical potentials above is almost due to two factors: (*a*) the probability of finding an inclusion of the most dangerous type (an oxide particle with a sulfide shell[2]) in the critical zone of the specimen (that is, the scratched or shielded area) and (*b*) the conditions of critical length for crevice corrosion for each inclusion.

The first factor controls the probability of nucleating a pit in the critical zone, so in our crevice corrosion tests this probability was larger than in the pitting tests, since the critical zone was larger.

In the second place, the dissolution of inclusions of the same shape in the different types of tests (that is, crevice corrosion or pitting tests) results in microcrevices of different lengths, because in crevice corrosion tests the microcrevice length is added to the external macrocrevice length, so the critical condition for propagation can be reached more easily. For these reasons we emphasize that the value of critical potential for crevice corrosion concerns only the defined experimental conditions.

Our statement that α'-martensite has little importance concerns only the case of our tests, in which the scatter in results does not allow us to draw a different conclusion, (though a beneficial effect of α'-martensite is sometimes possible, as in the case of the cold rolling).

A. I. Asphahani[3] (*written comment*)—To comment on the B. Syrett question: Why is the critical potential for crevice different from the critical potential for pitting (uncreviced specimens)? The active values of the critical pitting potential, if such a term can be used to describe results from tests on creviced specimens compared with those of uncreviced specimens, are presumably the result of a more acid/higher chloride content condition of the electrolyte within the crevice (even though the electrolyte outside the crevice is the same).

J. Kruger[4] (*written discussion*)—Would laser glazing (the rapid melting of a surface using a laser), which produces an amorphous metal surface, produce lower susceptibility to localized corrosion?

A. Cigada, B. Mazza, G. A. Mondora, P. Pedeferri, G. Re, and D. Sinigaglia (*authors' closure*)—It is possible, because we think inclusions are more dangerous than plastic deformation or phase transformation that sometimes appear to be beneficial.

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[3] Stellite Division, Cabot, Wokomo, Ind. 46901.

[4] National Bureau of Standards, Washington, D.C. 20234.

A. G. Hartline[5] (*written discussion*)—Was there any difference in attack between the deformed austenite and martensite in specimens deformed at liquid nitrogen temperature. That is, was pitting in either phase prominent?

A. Cigada, B. Mazza, G. A. Mondora, P. Pedeferri, G. Re, and D. Sinigaglia (*authors' closure*)—In our work we have not determined in which phase pits propagate. This determination seems very difficult to us, and it can be carried out only with different techniques, such as electron microscopy, because of the very small size of the α'-martensite platelets.

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[5] Al Tech Specialty Steel Corp., Dunkirk, N.Y. 14048.