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Effect of Environmental Variables on Localized Corrosion of High-Performance Container Materials

Ajit K. Roy
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Abstract

Electrochemical cyclic potentiodynamic polarization (CPP) experiments were performed on several candidate high-performance waste package container materials to evaluate their susceptibility to localized corrosion in aqueous environments relevant to the potential underground high-level nuclear waste repository. This paper presents the results of this study showing the effects of chloride ion (Cl^-) concentration, pH, temperature, and electrochemical potential scan rate on the pitting corrosion behavior of these materials.

Background

The current high-level nuclear waste package design is focused on all-metallic multi-barrier concepts to accommodate the nation's spent nuclear fuel and vitrified high-level nuclear waste for geologic disposal in the potential Yucca Mountain repository. This design incorporates a thick outer corrosion-allowance metal barrier over a thinner inner container made of corrosion-resistant alloy, so that the inner barrier may be galvanically protected should the outer barrier be breached.

Although the groundwater in the vicinity of the potential repository is believed to be non-aggressive for many corrosion-resistant alloys, under certain operating conditions the repository environments surrounding these waste packages could become very hostile. Under these conditions, the corrosion-resistant container might become susceptible to localized modes of degradation such as pitting and crevice corrosion.

It is well known(1-11) that environmental parameters such as Cl^- concentration, pH, and temperature can significantly influence the susceptibility of various metals and alloys to pitting corrosion. Furthermore, the potential scan rate can influence the electrochemical test results because of the variation in time needed in conducting the CPP tests, as cited(12-19) in the open literature. This paper presents the results of CPP experiments performed in many different aqueous salt solutions, showing the effects of Cl^- concentration, pH, temperature, and potential scan rate on the pitting and crevice corrosion susceptibility of several candidate waste package container materials.

Materials and Environments

Materials tested include iron-nickel-chromium-molybdenum (Fe-Ni-Cr-Mo) Alloys 825, G-3 and G-30; Ni-Cr-Mo Alloys C-4 and C-22; and titanium-base alloy Ti Grade-12. Their chemical compositions are given in Table 1. Test specimens were fabricated from solution-annealed materials by a qualified outside vendor, and additional thermal treatments were not given to these specimens prior to their exposure to the test environments.

Since the precise repository environment surrounding the waste packages is yet to be determined, tests were performed in deaerated neutral, acidic, and alkaline brines containing 1-10 weight percent (wt%) NaCl at 50°C, 70°C, and 90°C. Acidification was done by adding sulfuric acid to the brine; calcium hydroxide was added to prepare the alkaline salt solution. The pH for acidic, neutral, and alkaline brines ranged between 2 and 3, 6 and 7, and 10 and 11, respectively. A few tests were also performed in brines having slightly modified pH (4.36, 7.07 and 9.63). The rationale for selecting the concentrated, acidic, neutral, and alkaline brines as test environments has been provided elsewhere.(20)

Experimental Procedure

A three-electrode technique was used to perform CPP experiments in a Pyrex cell using a cylindrical test specimen as working electrode, two graphite counter electrodes, and a Luggin capillary connected to a reference electrode. Ag/AgCl was used as the reference electrode in all tests. The test specimens were polished with 600-grit paper and cleaned with distilled water, acetone and ethanol prior to their exposure to the test solutions.

Potential was applied to the test specimens at scan rates of 0.043, 0.085, 0.17 and 0.34 mV/sec using EG&G Models 273 and 283 potentiostats, controlled by an IBM-compatible PC with EG&G corrosion software. Forward potential scans were done in the noble direction commencing at the stable corrosion potential (E_{corr}), and continuing to a potential sufficiently noble to either cause pitting or exhibit pit-like indication prior to reversing the potential scan at the same rate.

A clockwise hysteresis loop is traced during reverse scan, indicating the possibility of pitting in susceptible alloys. Two pitting parameters, namely the critical pitting potential (E_{pit}), and the protection potential (E_{prot}) can be determined from these tests. The value of E_{pit} denotes the potential at which current increases abruptly on the forward scan, indicating the possibility of pit initiation. E_{prot} indicates the electrochemical potential at which the current returns to passive values during reverse scan, indicating repassivation of pits.

The pH of the test solution was measured at room temperature prior to initiation of each experiment. At the conclusion of each test, the specimen was cleaned with distilled water, acetone and ethanol. The cleaned specimen was visually examined, followed by an optical microscopic evaluation to detect the presence or absence of pitting.

Results and Discussion

Results indicate that Alloys 825, G-3, G-30 underwent pitting and crevice corrosion in all tested environments, with Alloy 825 exhibiting the maximum susceptibility (Figure 1). Alloy G-3 is comparable in composition to Alloy 825, but has increased Mo content for enhanced resistance to pitting corrosion. Alloy G-30 contains substantially more Cr and slightly higher Mo content than Alloy 825. Although the extent of pitting in Alloy G-30 was significantly less pronounced than that observed in Alloys 825 and G-3, it appears that the presence of higher Mo and Cr content alone cannot prevent the localized breakdown of the protective surface films on these three Fe-Ni-Cr-Mo alloys while exposed to 90°C concentrated brines under an electrochemical potentiodynamic control.

As to the localized corrosion susceptibility of Ni-Cr-Mo alloys, Alloy C-4 suffered from pitting corrosion in all tested environments. But the extent of pitting was less severe than that observed with Fe-Ni-Cr-Mo alloys tested in this study. The relatively reduced pitting tendency of Alloy C-4 can, possibly, be attributed to the presence of sufficiently higher Mo (15.38 wt%) and less Fe (0.77 wt%) contents. An interesting observation was that this alloy became susceptible to severe crevice corrosion in brines containing 5 and 10 wt% NaCl, as illustrated in Figure 2. With respect to the corrosion behavior of Alloy C-22, no pits were observed along the specimen surface by visual examination. However, optical microscopic evaluation at high magnifications revealed tiny pit-like dents along the specimen surface. Furthermore, this alloy showed slight crevice tendency and surface discoloration. But the specimen surface was still unattacked.

Neither pitting nor crevice corrosion was observed with Ti Grade-12 in any tested environment by visual examination. However, at very high optical microscopy magnifications, very few tiny pit-like dents were observed along the specimen surface.

Effect of NaCl Concentration

The initiation of pitting is the result of the breakdown of the passive film on the surface of metals and alloys in the presence of aggressive anions such as Cl^-, and the subsequent establishment of an electrochemical cell in which the damaged site acts as anode and the surrounding passive surface acts as a cathode. The relationship between E_{pit} and Cl^- concentration for all alloys tested in acidic brines, is illustrated in Figure 3. It appears that for Alloys 825, G-3, G-30 and C-4, E_{pit} was shifted to more active (negative) values with increasing Cl^- concentration. The effect of Cl^- concentration on E_{pit} values for these susceptible alloys appears to be consistent with that observed by other investigators.(1-8)

The concept of critical pitting potential, at or above which passive metals and alloys may become susceptible to pitting corrosion in the presence of halide ions, has been reviewed by Kolotyrkin(21), and Leckie and Uhlig.(8) Two theories have been proposed(8,22) to explain the transition from passivity to pitting in susceptible alloys. One of these theories, proposed by Hoar et al(22), suggests that pitting occurs as a result of adsorption of aggressive anions such as Cl^- on an oxide film, followed by penetration of this film under the influence of an electrostatic field. Pits initiate when the field across the film-solution interface reaches a critical value.

An alternate model(8) is based on reversible competitive adsorption of Cl^- into the oxide/liquid interface (double layer) with oxygen for sites on the alloy surface. Oxygen normally has higher affinity than Cl^- for an adsorption site, but as the electrochemical potential of the alloy is driven in the noble direction, more Cl^- ions move into the double layer. At a sufficiently high concentration corresponding to the critical potential (E_{pit}), Cl^- ions succeed at favored sites in destroying passivity by displacing adsorbed oxygen ions. The anodic overvoltage for dissolution of metallic ions is then appreciably reduced wherever the metal is in contact with Cl^- compared to metal in contact with adsorbed oxygen. Thus, metal ions rapidly enter solution resulting in a pit.

Figure 3 also illustrates the effect of Cl^- concentration on the critical potentials for Alloy C-22 and Ti Grade-12 in the acidic brines. It appears that, for Alloy C-22, increasing NaCl concentration from 1 to 5 wt% resulted in a shift of E_{pit} to a more active value. However, above 5-7 wt% NaCl, the critical potential was shifted to more noble values. For Ti Grade-12, E_{pit} was independent of Cl^- concentration. No clockwise hysteresis was observed for either Ti Grade-12 or Alloy C-22.

For Alloy C-22 and Ti Grade-12, which did not exhibit any pitting susceptibility, the critical potential became sufficiently noble to overlap the transpassive region, as shown in Figures 4 and 5, respectively. It has been suggested(23,24) that when the potential of a passivated anode is raised sufficiently, the potential drop across the double layer often becomes high enough for the oxidation of entities in the solution - often that of water, but, especially in concentrated solutions, also that of anions, cations or other dissolved substances. This may result in the formation of oxides on the alloy surface as anodic products, and corrosion of the electrode surface may then proceed uniformly with little or no localized corrosion. Such a phenomenon may possibly account for the absence of pitting attack in Alloy C-22 and Ti Grade-12 at the noble potentials corresponding to the pronounced rise of current density in the polarization diagrams.

Efforts were made to correlate Cl^- concentration to E_{corr} and E_{prot} values obtained from the CPP experiments. In many instances, E_{prot} could not be determined since the return polarization curve did not intersect the forward polarization curve at the passive current density, thus, showing no repassivation phenomenon in those tests. The results indicate that, for cases where E_{prot} was available, no consistent pattern on the effect of Cl^- concentration on this pitting parameter could be established. A similar statement can also be made for the effect of Cl^- concentration on E_{corr}.

Effect of pH

Figure 6 illustrates the effect of pH on E_{pit} for materials tested in brines containing 10 wt% NaCl. Data indicate that, for Alloys 825, G-3, G-30 and C-4, E_{pit} was shifted to more noble values with a change in pH from acidic to neutral. This observation matches the results of other investigators.(4,5) The more active E_{pit} value in acidic salt solution may be the result of acceleration of cathodic reaction due to high concentration of hydrogen ions. At alkaline pH, Alloys G-3, G-30 and C-4 showed slightly lower E_{pit} values compared to those in neutral solution. However, E_{pit} for Alloy 825 was shifted to a more noble value in alkalized brine. The more noble E_{pit} value for all four susceptible alloys at alkaline pH, compared to that in acidic brine, is consistent with the well-established pitting inhibition effect of hydroxyl ions.(25,26)

In contrast to the above observations, E_{pit} for Alloy C-22 was shifted to a more active value due to a change in pH from acidic to neutral, followed by a sharp rise at alkaline pH. For Ti Grade-12, E_{pit} was gradually moved to more active values with changing pH from acidic to neutral to alkaline. No explanation can presently be provided as to the cause of such behavior.

No consistent pattern on the effect of pH on E_{corr} and E_{prot} was observed.

Effect of Temperature

The relationship between E_{pit} and temperature for Alloys 825, G-3 and G-30 tested in acidic and neutral brines of various Cl^- concentrations is illustrated in Figures 7 through 12. In general, E_{pit} appears to shift to more active direction with increasing temperature, suggesting that these alloys may become more resistant to pitting corrosion at lower temperatures. The temperature dependence of E_{pit} appears to be consistent with that observed by other investigators.(3-6, 9,10)

These results may indicate the possibility of a temperature-induced change in the protective properties of the passive film on alloy surface , resulting in a reduction in its resistance to breakdown as temperature is increased. This change can be rationalized by taking two possibilities into consideration. First, the porosity of the passive film may increase with temperature, as often assumed. Second, the passive film may undergo an intrinsic modification of its chemical composition and/or physical structure resulting, for example, in a variation of the density of vacancies or voids in the oxide film. Based on existing data,(27,28) both possibilities should be considered.

An increase in porosity at increased temperature is supported by the fact that Cl^- ions have been found to incorporate into the passive film on stainless steel(27) at high temperatures, but not at lower temperatures. As to the second possibility, it has been reported(28) that the protective oxide film on stainless steel changes from a p-type at room temperature to an n-type at higher temperatures. However, not enough information currently exists on the effect of temperature on the morphology of passive films to decide which of these two alternatives is correct. It is possible that the observed relationship between E_{pit} and temperature may be attributed to a combined effect of both these factors.

Effect of Potential Scan Rate

The effect of electrochemical potential scan rate on E_{pit} for Alloys 825, G-3 and G-30 in 90°C acidic, neutral, and alkaline brines containing 10 wt% NaCl is shown in Figures 13-15. Results indicate that in the acidic and alkaline brines (Figures 13 and 15), E_{pit} was shifted to more positive values with an increase in scan rate from 0.043 to 0.085 mV/sec, followed by a drop (more anodic) at a faster scan rate of 0.17 mV/sec. In alkaline brines, E_{pit} was gradually moved to even more anodic values as the scan rate was doubled. However, in the acidic brine, a mixed behavior was observed. For Alloys 825 and G-3, the critical potential was shifted to more noble values at the fastest scan rate of 0.34 mV/sec. On the other hand, E_{pit} for Alloy G-30 became slightly more anodic at this scan rate.

As to the effect of scan rate on E_{pit} in the neutral brine, both Alloys 825 and G-3 exhibited a pattern which is similar to that observed in the acidic and alkaline brines at scan rates of 0.043, 0.085 and 0.17 mV/sec. But for Alloy G-30, just the opposite behavior was observed at these scan rates. At the scan rate of 0.34 mV/sec, however, a mixed pattern on the scan rate dependence of E_{pit} was observed, as shown in Figure 14.

It appears from the above results that the effect of potential scan rate on E_{pit} may vary with the alloys tested, and it is difficult to justify a general behavior. However, two basic types of E_{pit} response to increasing scan rate may be noticed for the alloy/environment combinations incorporated in this study. The first one is an initial shift in E_{pit} to more noble values with increasing scan rate followed by a shift in the active direction (Figures 13 and 15). A similar shift in E_{pit} in the active direction with increasing scan rate has been reported by Bond and Lizlovs(12), which is in contrast to the observation by Leckie(19) who reported more noble E_{pit} values at faster scan rates. The second type may consist of an initial shift of E_{pit} in the noble direction in response to a faster scan rate followed by subsequent shifts in the active and noble directions, respectively (Figures 13 and 14).

It has been suggested(12,14,15,18,19) that induction time is necessary for pit formation because the passive film should be thinning prior to the initiation of pits. However, differences in opinion exist as to the role of induction period on pit initiation mechanism. At faster scan rates,(14,15,18,19) the exposure time may be too short to cause premature breakdown of the passive film on the alloy surface. But at slower scan rates, longer exposure times may permit pit initiation at a more negative potential during the forward scan. On the other hand, an alternate explanation has been proposed(12) to account for an ennoblement of pitting potential at slower scan rates. Such an explanation can possibly be rationalized by assuming that the passive film may thicken with time, at a potential within the passive region. Thus, the passive film would become thicker the slower the speed of scanning. It is difficult to speculate which of these two alternatives is technically more viable.

Summary and Conclusions

CPP experiments were performed in concentrated, acidic, neutral, and alkaline brines to evaluate the susceptibility of Alloys 825, G-3, G-30, C-4, C-22, and Ti Grade-12 to localized corrosion. The effect of Cl^- concentration, pH and temperature on E_{corr}, E_{pit} and E_{prot} was investigated. The relationship between E_{pit} and electrochemical potential scan rate was also analyzed. The significant conclusions drawn from this study are the following:

- Alloys 825, G-3 and G-30 became susceptible to pitting and crevice corrosion in all environments tested under potentiodynamic control.

- Alloy C-4 suffered from pitting and crevice corrosion in acidic, neutral, and alkaline brines at 90°C. But the extent of damage was less pronounced compared to that of the Fe-Ni-Cr-Mo alloys.

- Alloy C-22 and Ti Grade-12 were immune to pitting corrosion under all experimental conditions used, thus demonstrating their suitability.

- For alloys susceptible to pitting, E_{pit} was shifted to more active values with increasing Cl^- concentration, confirming observations by other investigators.

- For Alloy C-22 and Ti Grade-12, which showed sufficiently noble critical potential to overlap the transpassive region, formation of protective oxides on alloy surface resulting from oxygen evolution due to oxidation of test solutions may possibly account for enhanced pitting resistance.

- In brines containing 10 wt% NaCl, E_{pit} for susceptible alloys was shifted to more noble values due to a change in pH from acidic to neutral. At alkaline pH, Alloys G-3, G-30 and C-4 showed somewhat lower E_{pit} values compared to those in neutral brines. For Alloy 825, E_{pit} was shifted to a slightly more noble value in alkalized brine.

- The more active E_{pit} value for susceptible alloys in acidic salt solutions may be the result of acceleration of cathodic reaction due to high concentration of hydrogen ions. The inhibitive effect of hydroxyl ions may possibly account for more noble E_{pit} value at alkaline pH.

- Consistent with results of other investigators, E_{pit} was found to become more active with increasing temperature, suggesting the occurrence of a temperature-assisted change in properties of protective surface films.

- With respect to the effect of potential scan rate on E_{pit}, a general trend was not observed that would be valid for all alloy/environment combinations studied. E_{pit} response to scan rate appears to be a function of the kinetics of passive film formation at applied potentials.

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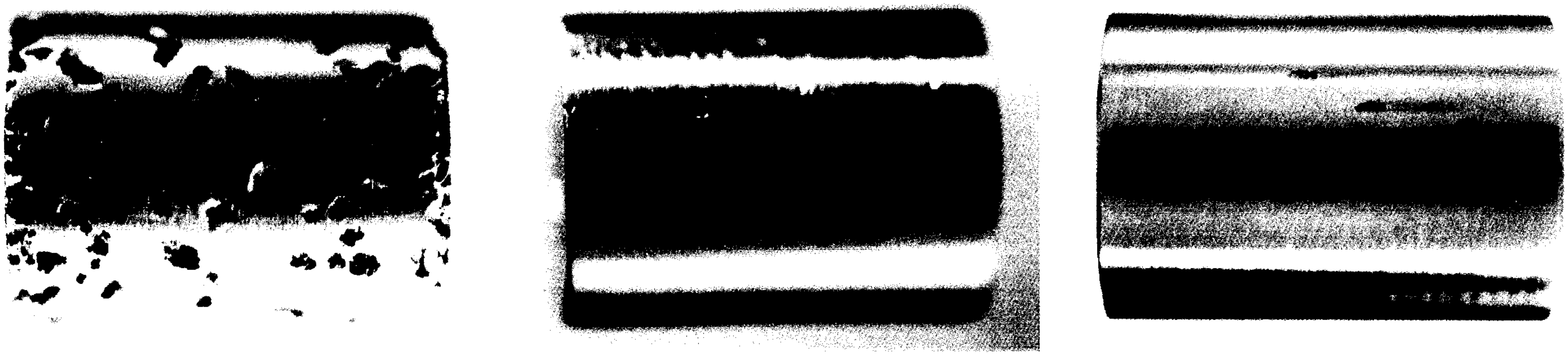
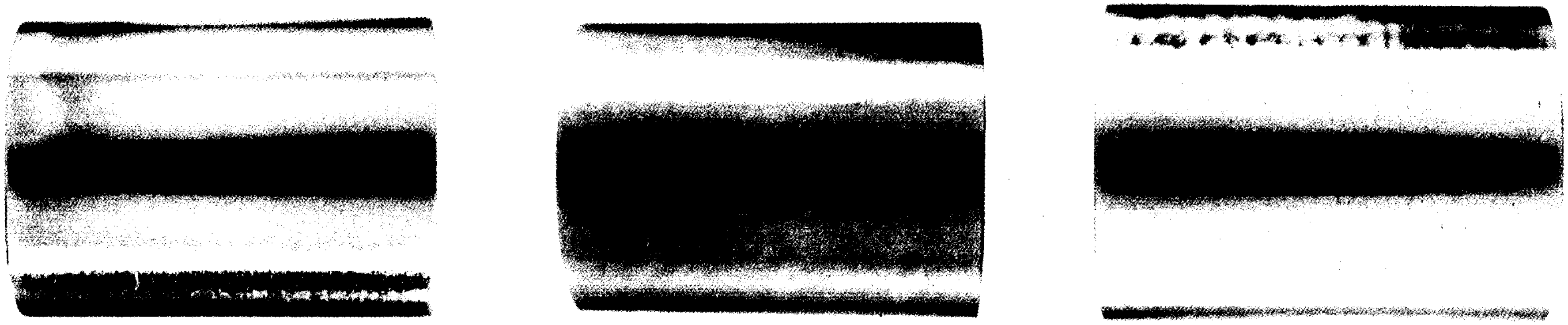
Table 1

Chemical Composition of Materials Tested (wt%)

Material	Heat No.	C	Mn	P	S	Si	Ni	Cr	Mo	Fe	Ti	Al	Cu	Others
Alloy 825	L256	0.01	0.42	-	<0.001	0.26	Bal	22.93	2.71	27.74	0.98	0.09	1.73	-
Alloy G-3	D495	0.01	0.47	0.012	0.005	0.03	Bal	22.70	6.88	19.60	-	-	1.96	-
Alloy G-30	L466	0.01	1.14	0.012	0.002	0.48	Bal	28.96	5.39	14.11	-	-	1.88	Nb+Ta: 0.79 Co: 3.18 W: 2.85
Alloy C-4	B033	0.003	0.21	<0.005	< 0.002	0.04	Bal	15.68	15.38	0.77	0.24	-	-	-
Alloy C-22	J244	0.004	0.21	0.004	<0.001	0.03	Bal	20.65	14.08	2.61	-	-	-	-
Ti Grade-12	H427	0.012	-	-	-	-	0.80	-	0.30	0.14	Bal	-	-	-

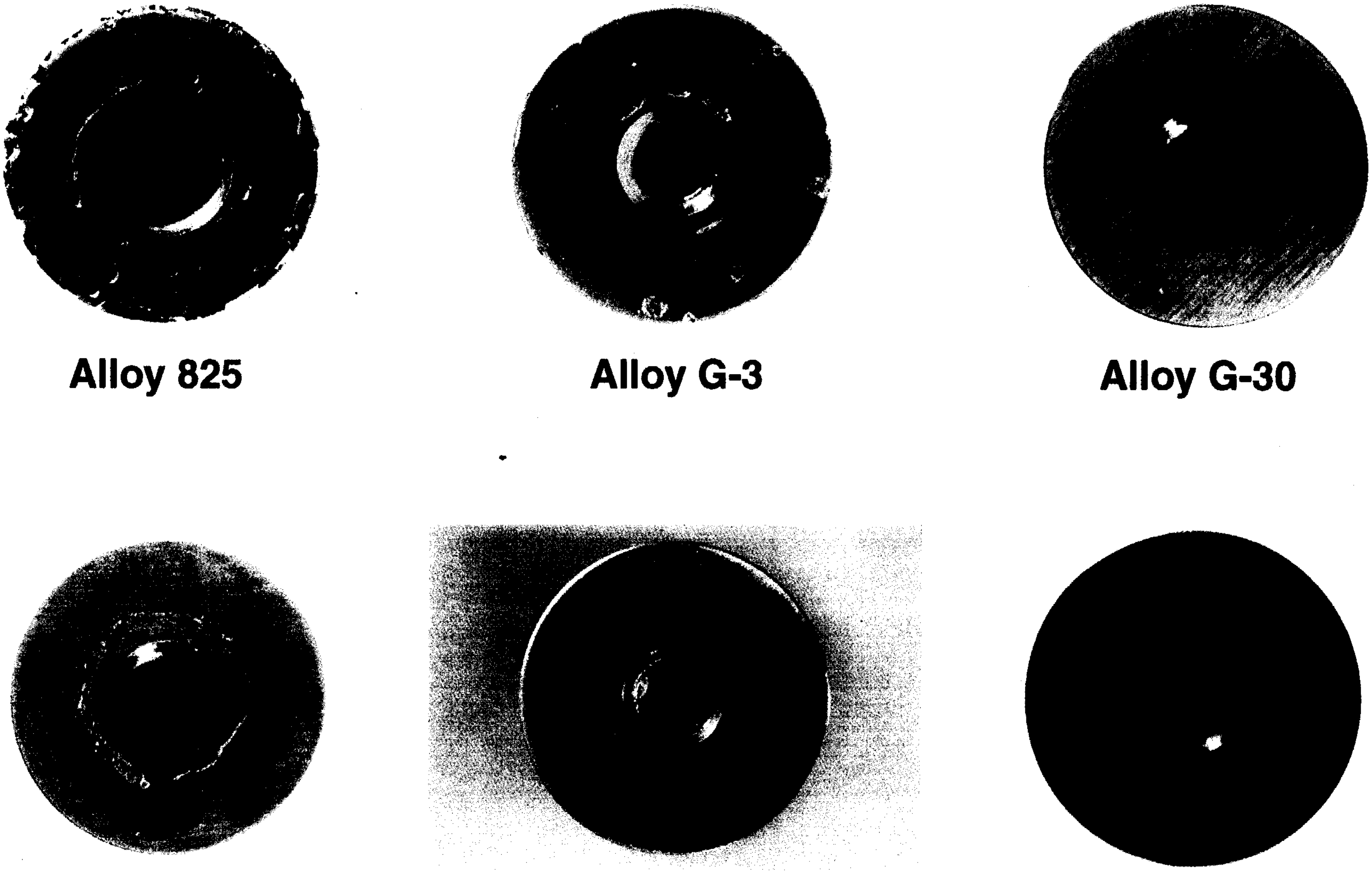


Alloy 825 **Alloy G-3** **Alloy G-30**



Alloy C-4 **Alloy C-22** **TiGr-12**

Figure 1. Surface Appearance (Horizontal Mode) of Materials Tested in Acidic Brines Containing 10 wt% NaCl at 90°C



Alloy 825

Alloy G-3

Alloy G-30

Alloy C-4

Alloy C-22

TiGr-12

Figure 2. Surface Appearance (Vertical Mode) of Specimens Tested in Acidic Brines Containing 10 wt% NaCl at 90°C

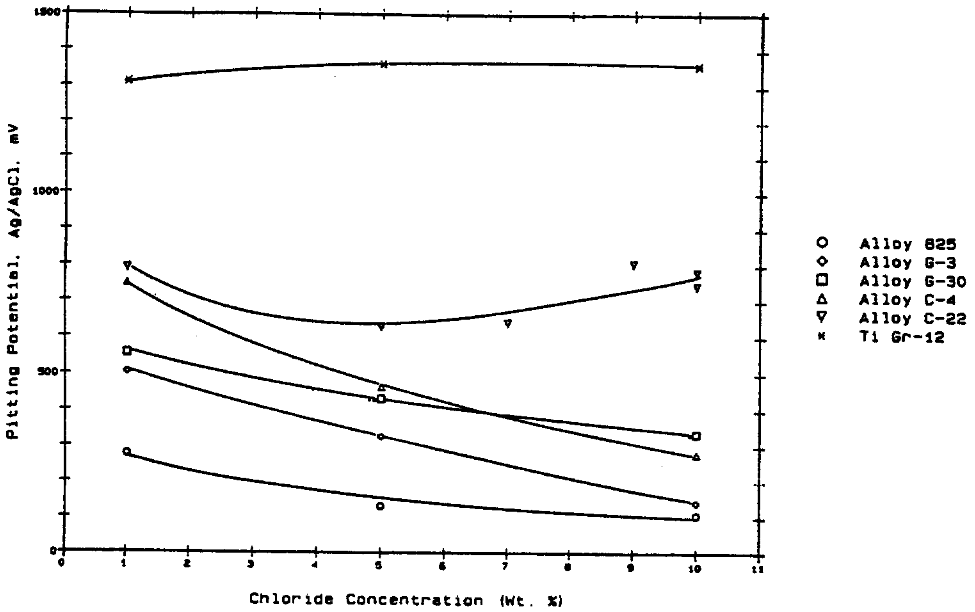


Figure 3. Pitting Potential vs Chloride Concentration in Acidic Brines at 90°C

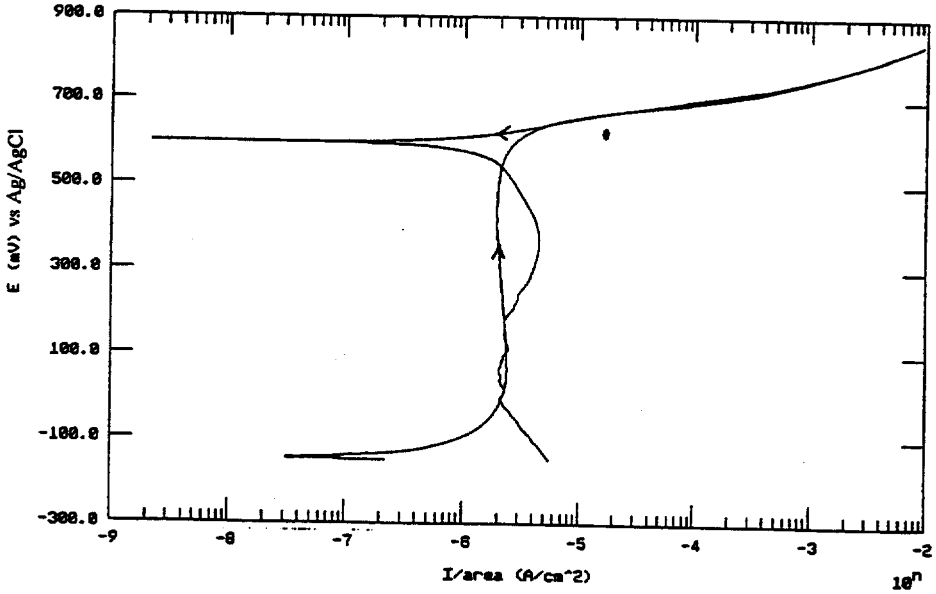


Figure 4. CPP Diagram of Alloy C-22 in Acidic Brine Containing 5 wt% NaCl at 90°C

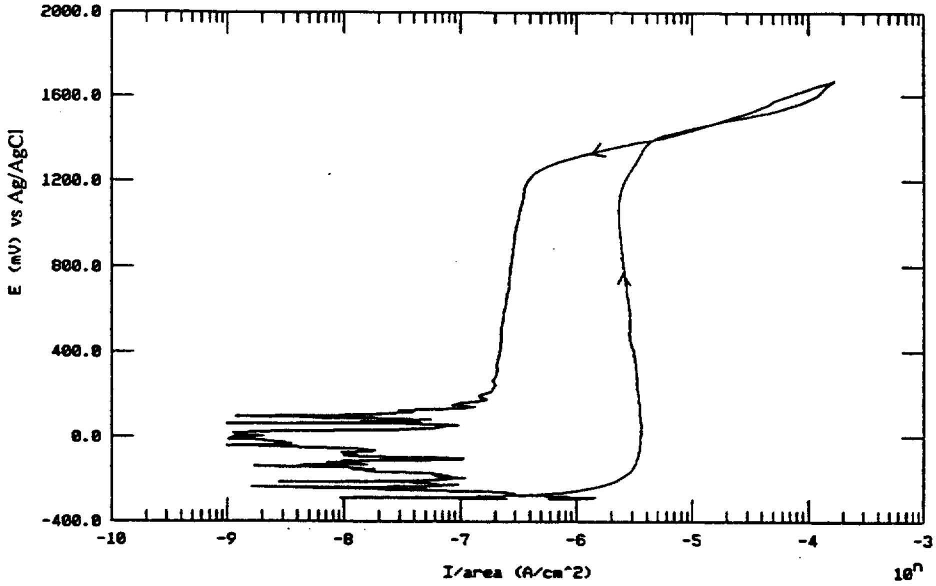


Figure 5. CPP Diagram of Ti Grade-12 in Acidic Brine Containing 5 wt% NaCl at 90°C

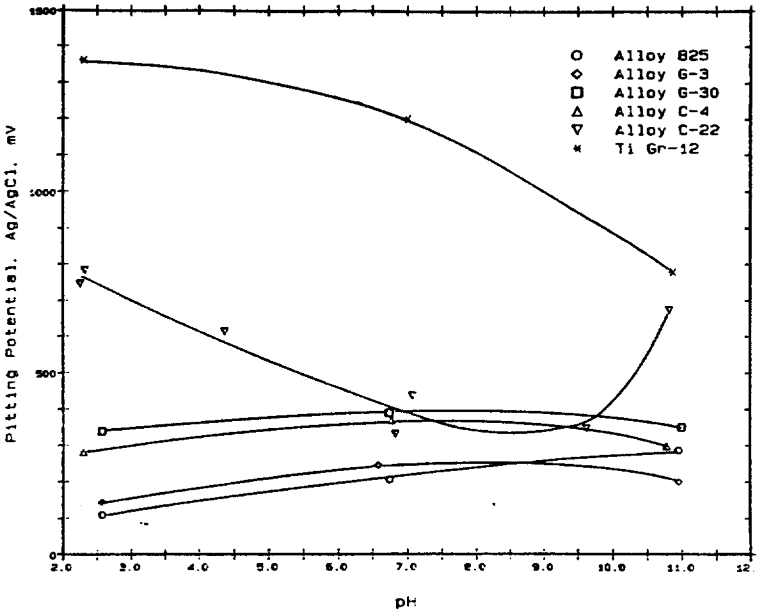


Figure 6. Pitting Potential vs pH in Brines Containing 10 wt% NaCl at 90°C

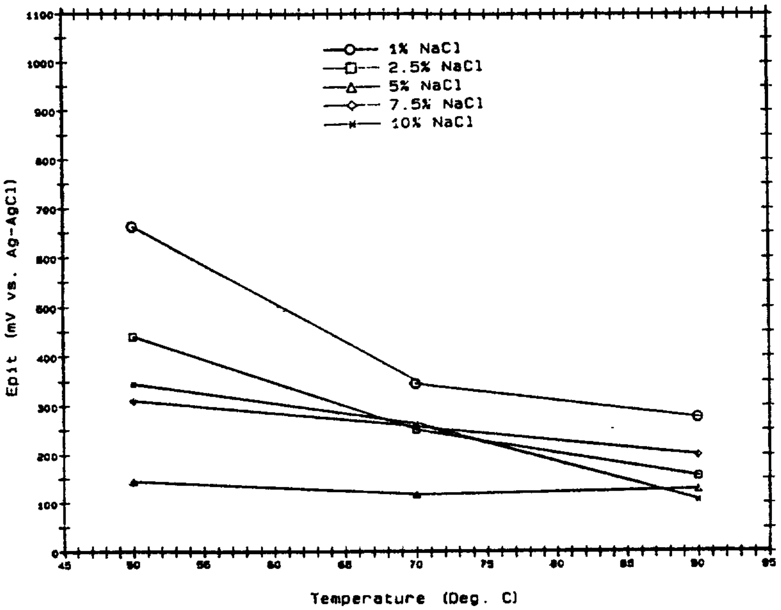


Figure 7. Pitting Potential vs Temperature for Alloy 825 in Acidic Brines

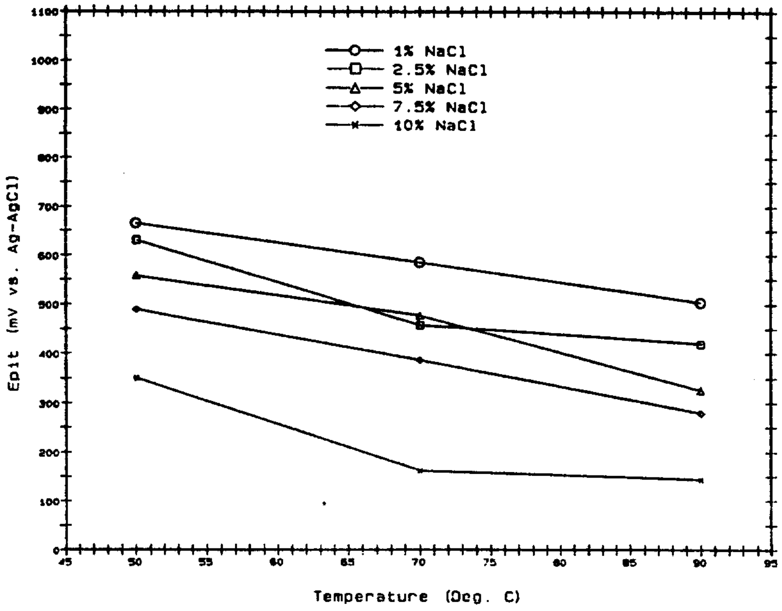


Figure 8. Pitting Potential vs Temperature for Alloy G-3 in Acidic Brines

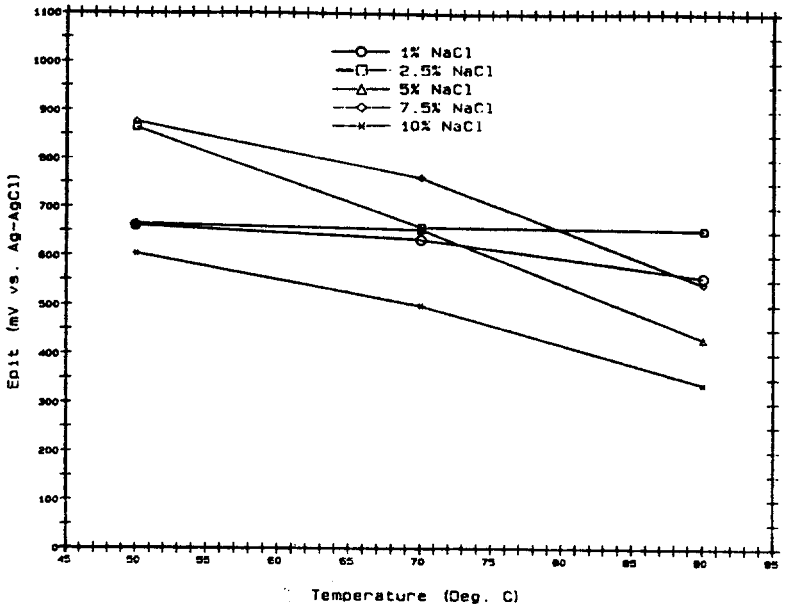


Figure 9. Pitting Potential vs Temperature for Alloy G-30 in Acidic Brines

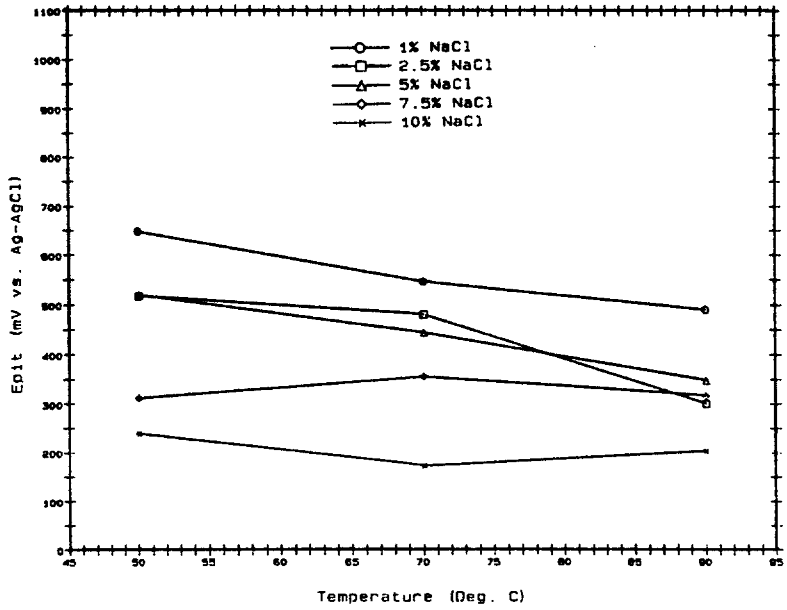


Figure 10. Pitting Potential vs Temperature for Alloy 825 in Neutral Brines

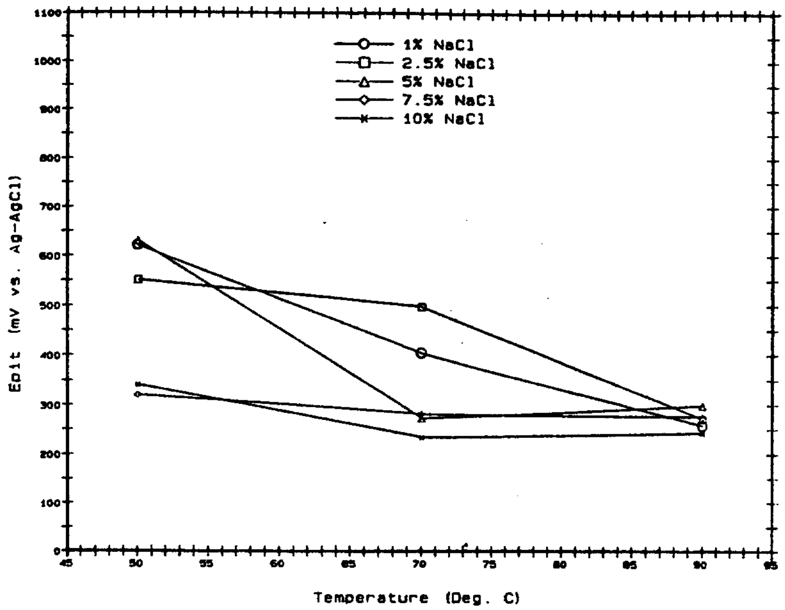


Figure 11. Pitting Potential vs Temperature for Alloy G-3 in Neutral Brines

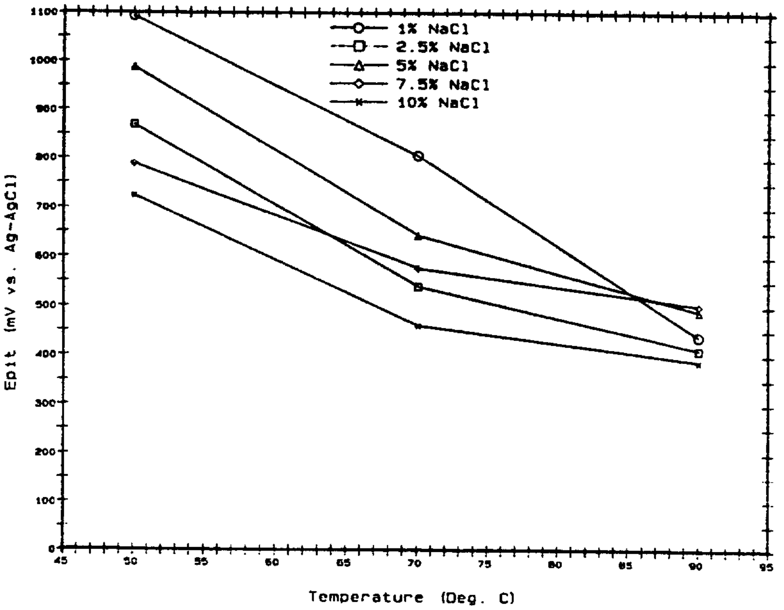


Figure 12. Pitting Potential vs Temperature for Alloy G-30 in Neutral Brines

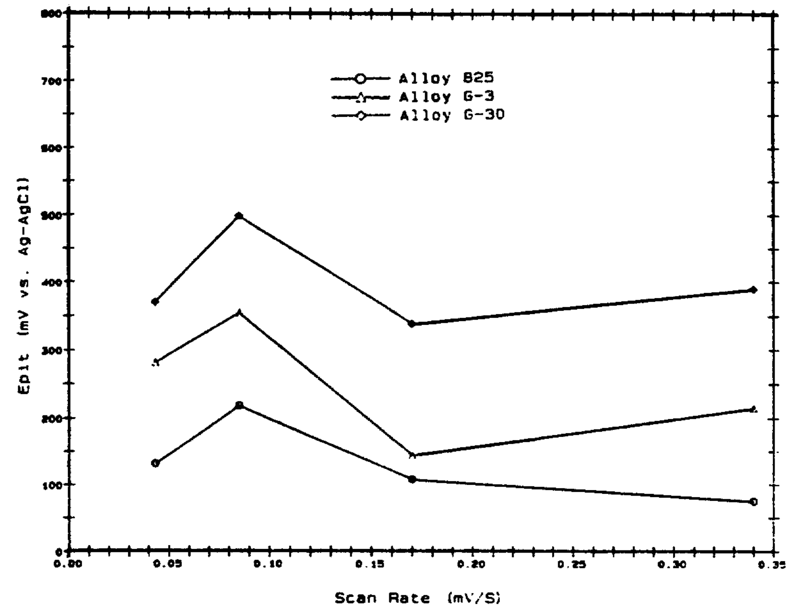


Figure 13. Pitting Potential vs Scan Rate in Acidic Brines Containing 10 wt% NaCl at 90°C

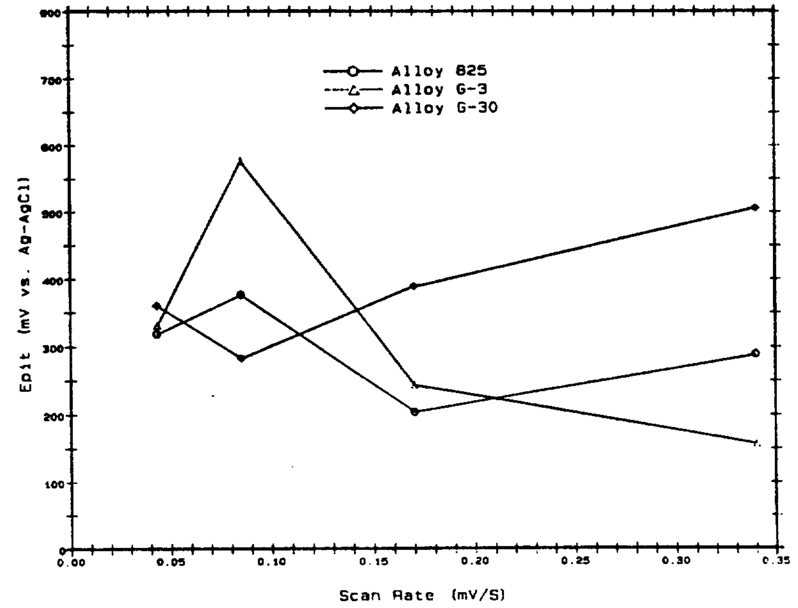


Figure 14. Pitting Potential vs Scan Rate in Neutral Brines Containing 10 wt% NaCl at 90°C

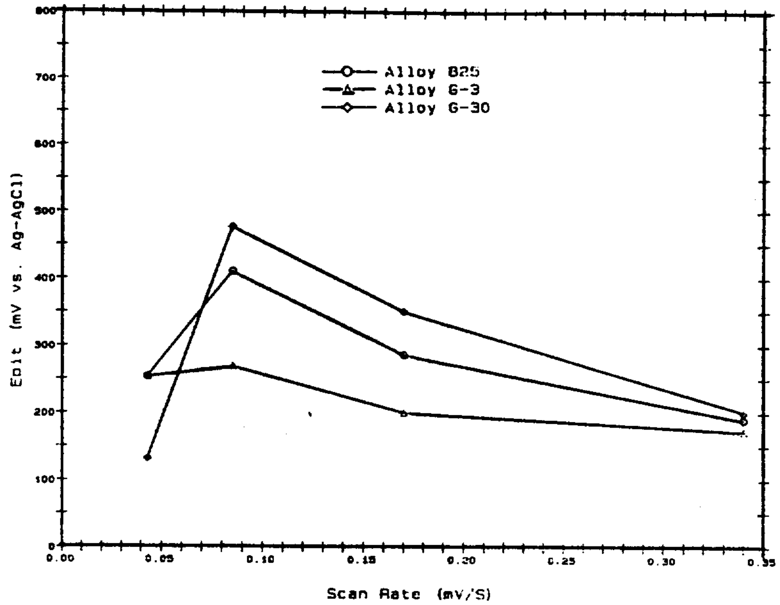


Figure 15. Pitting Potential vs Scan Rate in Alkaline Brines Containing 10 wt% NaCl at 90°C

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