

Ceramic to metal sealings: interfacial reactions mechanism in a porcelain–kovar junction

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Abstract

Several processes occur during the sealing of preoxidized kovar with porcelain through a glassy interphase. In the kovar, the strongly perturbed superficial zone tends to homogenize in composition as well as in the porosity distribution. The oxide scale is rapidly dissolved by the glass but the mechanism is complex with two elementary steps: (1) the diffusion of iron into the glass; (2) the devitrification of the glass enriched in iron (and may be in cobalt) to form a fayalite-like phase (type Fe_2SiO_4); however, crystallization is slower than iron diffusion so that, for well-chosen times and temperatures, the oxide scale is entirely dissolved into the glass before any devitrification. The glassy phase then penetrates into the open porosity of the alloy and a solid and helium-tight junction is achieved in this way. The iron-rich glass forms a “buffer zone” with a thermal expansion coefficient near that of the underlying alloy, but if the dissolved iron oxide layer is not thick enough, then the dilatometric behaviours of glass and alloy remain unadapted and the bonding breaks. The main knowhow of this kind of sealing consists in the good suitability of preoxidation of kovar with the characteristics of the thermal treatment for sealing.

Keywords: Ceramics; Metals; Glasses; Bonding; Sealings

1. Introduction

Ceramic to metal sealings, processed by means of a glassy interlayer, may lead to solid helium-tight junctions between porcelain and kovar [1]. It is easy to achieve a good interfacial bonding between porcelain and the glassy phase (enamel), well known in china-ware. The difficulties lie in the alloy to enamel interface. First, enamel must wet the alloy; at this stage the major role is played by iron monoxide which makes the glass more fluid and more wetting [2]. Thus it is necessary to preoxidize the alloy (to form the iron oxide), and this first step must be well controlled [3]. Then, as the thermal treatment requires temperatures of about 1000 °C, the dilatometric behaviours of the two materials in the interfacial zone must be very close, to avoid giving rise to stresses disastrous for the junction after cooling [4].

The quality of the junction strongly depends on the control of three main parameters: the thickness of the oxide scale formed on kovar by the preoxidation and the duration and the temperature of the sealing thermal treatment [1]. From that, it would be very interesting to know what are the features of the “ideal interface” obtained when these three parameters are well fit. Unfortunately only a few papers are available on this topic [5–10] and their definition of the “ideal interface” is different. For some of them [5–7] a solid junction necessitates that the oxide scale should be fully dissolved, except a monolayer, while others [8–10] consider that a layer of iron oxide must remain to give good mechanical properties to the junction. Furthermore, no dynamic reactional mechanism is given to explain the morphology of the interfacial zone.

For these reasons, we developed our study in this direction, following, first, the behaviour of preoxidized alloy during the sealing treatment and, second, the interfacial reaction. The alloy used is kovar (Fe,

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54 wt.%; Co, 19 wt.%; Ni, 27 wt.%), which is frequently employed in glass to metal junctions.

2. Experimental

2.1. Kovar samples

The kovar samples are small discs (diameter, 10 mm; thickness, 0.9 mm) of mass about 580 mg, cut with a diamond saw from a cylindrical rod provided by Imphy SA (Imphy, France) with the reference 08D1006. The main characteristics of this alloy, given by the supplier, are reported in Table 1. No decarburizing treatment was performed because of the very weak content of carbon.

The kovar–porcelain sealing cannot be carried out in air: the alloy would be strongly damaged, considering the temperature of the thermal treatment [3]. As far as it is necessary for the junction to have an oxide scale on the alloy [2,11], but in order to control its thickness, a preoxidation treatment in air is first performed, before achieving the sealing under flowing argon. From Zanchetta [1] a preoxidation treatment good for bonding includes an heating to 700 °C (with a rate of 25 K min⁻¹), maintained for 5 min, and followed by cooling at the same rate (25 K min⁻¹). We keep these conditions.

2.2. Porcelain discs

Porcelain discs (diameter, 20 mm; thickness, 4 mm) are obtained by slip casting in a plaster mould. The slurry is made of 1 kg of porcelain paste (quality CM10), supplied by KPCL SA (Limoges, France) mixed with 470 cm³ of water. The composition of the porcelain paste, according to KPCL SA, is given in Table 2.

The green pieces are fired in air at 1280 °C and covered after cooling, by an enamel paste provided by CERDEC SA (Limoges, France), the composition of which has been published elsewhere [2]. Discs are dried at 100 °C for 30 min and heated at 910 °C in air,

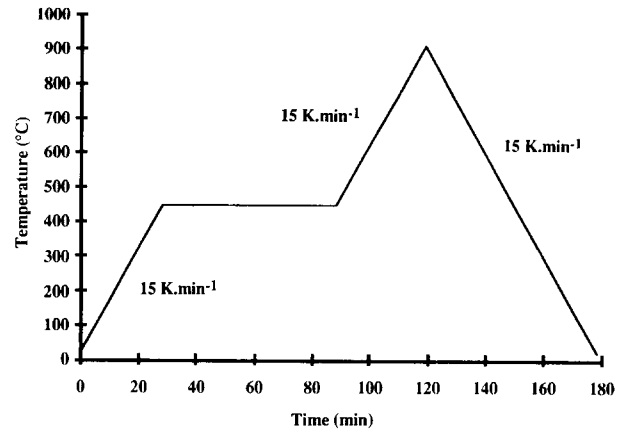


Fig. 1. Thermal treatment for enamelling.

following the thermal cycle described in Fig. 1 [1]. It includes a hold of 60 min at 450 °C for burning the binder of the enamel paste.

2.3. Sealing

Sealing tests are carried out in an alumina vertical tube furnace and the temperature is measured with a thermocouple located just below the sample. The oxygen content may be controlled by an electrochemical probe.

The following bonding conditions are again taken from Zanchetta's work [1]. The kovar disc is put on the enamelled porcelain piece and they are first heated under vacuum (10 Pa) to 300 °C and then under flowing argon (containing less than 5×10^{-4} vol.% O₂ and 5×10^{-4} vol.% H₂O). After the thermal cycle of Fig. 1, the soaking temperatures of 910 or 940 °C are reached at a rate of 15 K min⁻¹ except for the last 20 or 30 K where the rate is reduced to 3 K min⁻¹. After a time varying from 3 to 60 min, samples are rapidly cooled to 620 °C, then slowly to 550 °C where they are annealed for 15 min. These parameters were determined on the basis of Zarzyki's work [12] and considering the characteristics of the enamel (softening and glass transition temperatures). The rate of final cooling is low (3 K min⁻¹) to minimize the strains.

Table 1
Characteristics of kovar

Composition (wt.%)							Density (g cm ⁻³)
Fe	Ni	Co	Mn	Si	C		
53.19	29.35	17.04	0.27	0.15	0.0033	8.25	

Table 2
Composition (weight per cent) of the porcelain CM10

SiO ₂	Al ₂ O ₃	K ₂ O	CaO	Na ₂ O	MgO	Fe ₂ O ₃	TiO ₂	Firing loss
60.35	25.85	3.40	0.79	0.63	0.15	0.42	0.12	8.29

For the observations and analysis of the interfacial zone of the bonded samples, they are wrapped up in a resin (to avoid any unsticking), cut with a diamond saw and then properly polished.

3. Influence of the sealing treatment on the preoxidized kovar

After preoxidizing, the pieces of kovar are covered by a complex oxide scale composed [3], from outside to inside, of an external layer of hematite Fe_2O_3 , a layer of magnetite Fe_3O_4 with a low lattice parameter (0.8364 ± 0.0003 nm) and a complex zone where magnetite coexists with an alloy enriched in nickel and cobalt, which constitutes the very irregular oxide–alloy interface. All these layers are porous and mechanically brittle; they are presented in Fig. 2, for a sample obtained as described before (Section 2.1).

Because the sealing treatment requires temperatures distinctly higher than that of preoxidation, and with a different atmosphere (argon instead of air), it is important to examine firstly the modification appearing in the alloy and the oxide scale before the beginning of the interfacial bonding reaction.

3.1. Experimental results

To enhance better the temperature effect, the observations concern not only the range 900–1000 °C, where the bonding is achieved, but also higher temperatures up to 1300 °C. Samples undergo a heating under argon in the same conditions as for bonding (Section 2.3) but as soon as the required temperature is reached they are quenched in argon in order to keep the morphologies and structures present at the time of bonding.

Metallographical observations of Fig. 3 and scanning electron microscopy (SEM) micrographs of Fig. 4



Fig. 2. Oxide scale on preoxidized kovar.

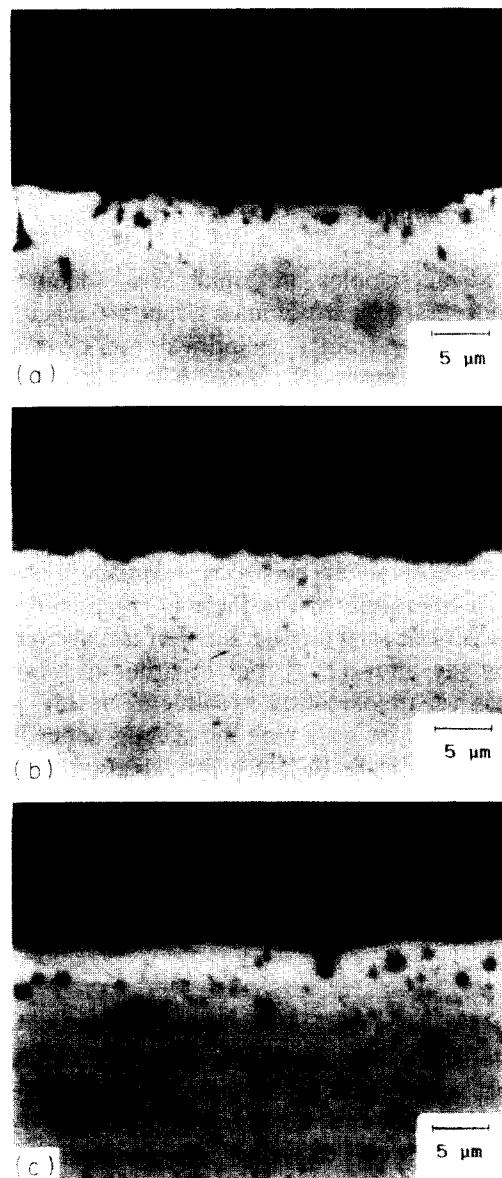


Fig. 3. Metallographical observations of cross-sections of preoxidized kovar heated and quenched in argon at (a) 900 °C, (b) 1000 °C and 1300 °C.

show, by comparison with Fig. 2, that temperature has an important morphological effect on the oxide scale and on the inner alloy.

Between 900 and 1000 °C the complex interfacial zone tends to form a more regular and well-defined interface between oxide and alloy (Figs. 3(a) and 3(b)). Fig. 3(b) shows that a thin porosity and some grey spots appear in the alloy, coming from the porous perturbed interfacial zone visible in Fig. 2, while Fig. 4(a) indicates that the oxide scale becomes clearly more compact and no longer has the two distinct areas of Fig. 2. These two effects are amplified at 1300 °C: the porosity in the alloy is even higher (Fig. 3(c)) and the oxide scale more compact (Fig. 4(b)) than at lower

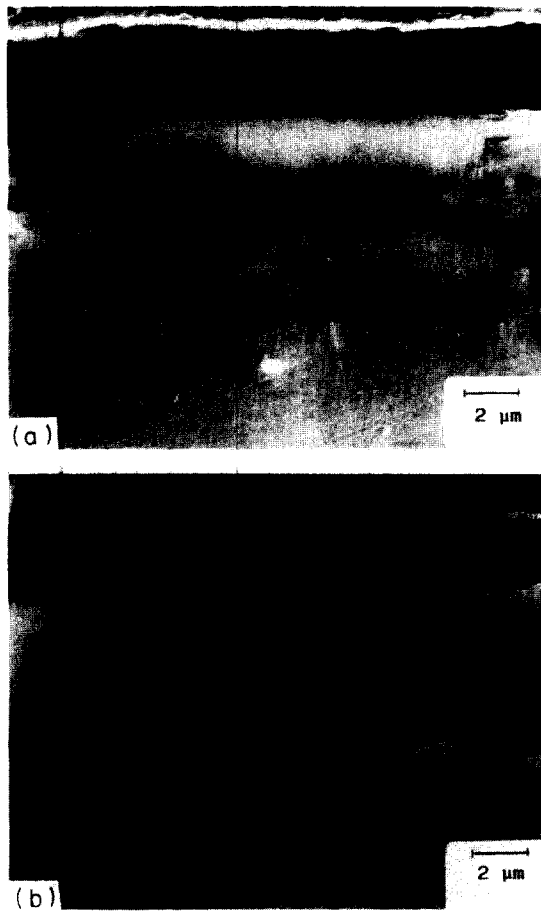


Fig. 4. SEM micrographs of the kovar–oxide interface of preoxidized pieces treated and quenched in argon at (a) 900 °C and (b) 1300 °C.

temperatures, when the presence of a secondary grey phase becomes obvious, with small spherical inclusions.

X-ray diffractometry of the surface of the pieces shows that the oxide scale is modified from 800 °C. At this temperature, the hematite phase disappears, being replaced by a wüstite-like phase containing perhaps some cobalt. The Fe_3O_4 spinel phase remains, but its lattice parameter grows from 0.8364 ± 0.0003 nm to 0.8388 ± 0.0003 nm (Table 3). Also, the structure of the underlying alloy “seen” through the oxide scale is

appreciably modified. It is no longer composed of the usual two phases of kovar (iron (α -Fe) and taenite (Fe, Ni) with cobalt) but of only one cubic phase with the same structure as Ni_3Fe (awaruite), certainly containing cobalt.

At 900 °C, the spinal phase has almost completely given way to the wüstite-like phase whose lattice parameter is 0.4287 ± 0.0003 nm when the X-rays of the cubic alloy slowly shift towards the small angles.

At 1000 °C the only oxide scale is the wüstite-like phase and the alloy structure becomes intermediate between those of awaruite and taenite (NiFe).

For higher temperatures, the lattice parameter of the wüstite-like phase slowly grows and the alloy has the taenite structure.

In short, the oxide scale goes from a complex and porous structure, at 700 °C, with Fe_2O_3 and Fe_3O_4 phases, to a monophasic and rather compact FeO scale (with maybe cobalt traces) at 1000 °C.

At the same time, the porosity of the interfacial zone, accompanied by some inclusions, spreads to the inner alloy whose structure, initially diphasic, becomes monophasic, firstly of the awaruite type and then the taenite type.

(EDAX) profiles of cross-sections of some quenched pieces confirm these evolutions. At 700 °C (Fig. 5(a)) [3], one can see that the external oxide layer only contains iron oxides and that the inner oxidation zone is strongly impoverished in iron and hence, logically, enriched in cobalt and nickel. As the temperature grows, there is no appreciable migration of cobalt or nickel in the oxide scale during the structure changes at 900 °C (Fig. 5(b)) or, higher, at 1300 °C (Fig. 5(c)). On the contrary, significant homogenization of the alloy occurs, with a widening of the iron-impoverished zone which becomes less poor in iron, and correlatively less rich in cobalt and nickel. These changes are consistent with the structural modifications observed by X-ray diffractometry.

The EDAX analysis of the small inclusions which migrate with porosity inside the alloy towards its center (Fig. 6(a)) shows that they are composed of iron oxide as the only peaks detected are those of oxygen and iron (Fig. 6(b)).

Table 3
Lattice parameter of crystalline phase observed after argon quenching

Oxide phase	Lattice parameter (nm)					JCPDS–ICCD value
	Value for the following quenching temperatures (°C)					
	800	900	1000	1200	1300	
Magnetite (Fe_3O_4)	0.8388 ± 0.0003					0.8396
Wüstite (FeO)		0.4287 ± 0.0003	0.4289 ± 0.0003	0.4294 ± 0.0003	0.4296 ± 0.0003	0.4303

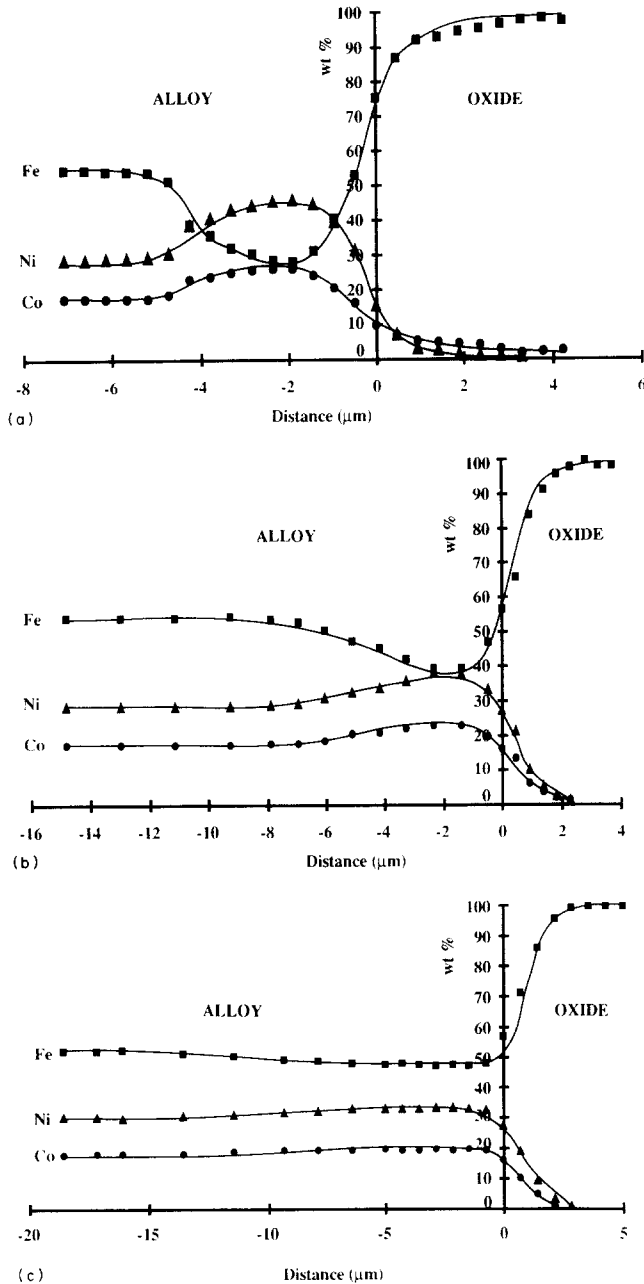


Fig. 5. Evolution of profiles of concentration of iron nickel and cobalt: (a) kovar preoxidized at 700 °C from Ref. [3]; the same heated then quenched (b) at 900 °C and (c) at 1300 °C.

3.2. Discussion

3.2.1. Behaviour of the preoxidized alloy below 900 °C

When the temperature increases, at the beginning of the sealing treatment, the biphasic alloy near the interface becomes poorer in iron, since we observe the disappearance of the phase of type taenite (Ni, Fe) and its replacement by awaruite type (Ni₃Fe) at 800 °C. This effect is probably due to the transformation of the hematite and of the spinal phases of the oxide scale into wüstite, accompanied by an outward

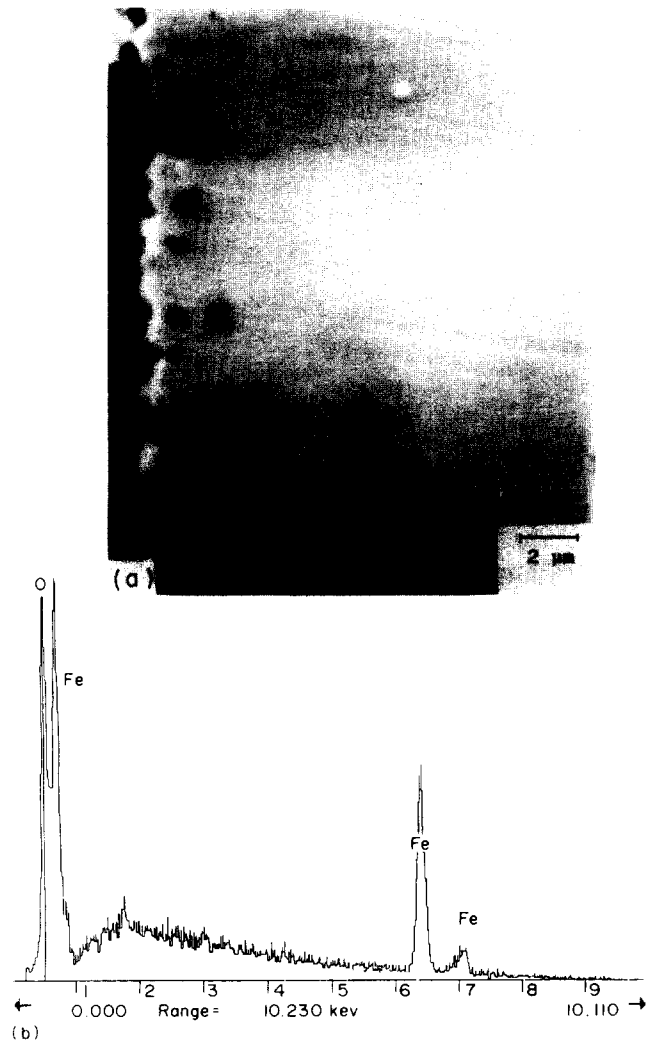


Fig. 6. Observations and analysis of alloy quenched at 900 °C: (a) small inclusions (A, B, C) and porosity (D, E, F); (b) EDAX spectrum of the small inclusion C.

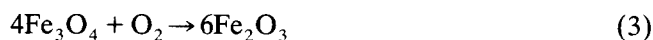
diffusion of iron, already observed during the oxidation of kovar [3]. This iron, drawn from the underlying alloy substrate, cannot be immediately replaced by centrifugal diffusion in the alloy, from which the noticed impoverishment results.

Considering the presence of cobalt in the alloy, its composition near the interface can be formulated by (Ni_{1-x}Co_x)₃Fe during the rise of temperature, in the approximate range 800–900 °C and for the considered increasing rate of 15 K min⁻¹.

In the oxide scale, a change of phase occurs, due to the iron diffusion. First the hematite phase, and, second, the magnetite phase are reduced, i.e.



both of these being in competition with the outermost reaction at the Fe₃O₄-O₂ interface, i.e.



owing to the persistence of oxygen traces in flowing argon. However, X-ray analysis shows that reactions (1) and (2) are preponderant.

The growth of the lattice parameter of the spinal phase shows that its composition tends towards that of pure magnetite (cf. Table 3). This can be explained by the inner reduction of the magnetite-type phase: only the X-ray diffraction peaks of the magnetite coming from the outer part of the oxide scale remain, resulting from the reduction of the hematite which never contains any cobalt. Moreover, a weaker non-stoichiometry, as a result of the diffusion of iron, may also explain this growth of the lattice parameter.

3.2.2. Temperatures higher than 900 °C

For temperatures higher than 900 °C, the growth of the diffusion rate of iron from the alloy bulk towards the kovar–oxide interface involves a less marked impoverishment in iron of the alloy near the interface. From that, there is an evolution of the cubic phase and a beginning of migration of the pores towards the bulk: the alloy becomes more homogeneous. Inside the oxide scale, the iron diffusion effect favours reaction (2) to the detriment of reaction (3), reaction (1) being over.

At 1000 °C, the oxide scale is entirely reduced into a wüstite-like phase, but this phase may still react, on the outer interface, with the oxygen traces of the flowing argon by



So, reactions (2) and (4) are in competition, but X-ray analysis confirms that reaction (2) is preponderant, and all the more so since the diffusion rate of iron increases in the alloy and in the oxide scale. This increase in the iron diffusion rate in the alloy finally leads to the following.

(1) First, there is an acceleration of the migration of the pores towards the inner alloy, which homogenizes with a widening and a filling of the Fe depletion area; at 1300 °C, the composition of the outer alloy is soon different from that of inner alloy owing to the lack of the iron phase. At the same time small inclusions of iron oxide appear in the alloy near the oxide interface and then spread in the kovar.

(2) Second, there is a growth in the lattice parameter of the wüstite phase (Table 3) with a decrease in its non-stoichiometry.

It is to be noticed that cobalt and nickel do not diffuse (or very little) in the oxide scale. From Macey et al. [13], cobalt diffusion is favoured by high oxygen pressures; the low oxygen content (about 2.5 Pa) that we have here may explain the lack of cobalt diffusion.

4. Evolution of the interfacial zone during sealing

After following the behaviour of the metallic part alone during the bonding thermal treatment, we now consider the modifications occurring in the glassy interphase during sealing.

4.1. Influence of time

For this study, the temperature of 910 °C is chosen and, following the thermal cycle described in Fig. 1, the soaking time varies from 3 to 60 min.

The metallographical observations for the shorter time, i.e. 3 min (Fig. 7(a)), show that the behaviours of the alloy and of the oxide scale are in accordance with the previous results of Section 3: the oxide pockets and the porosity diffuse towards the inner alloy, when the oxide scale tends to homogenize. However, a gradation of grey appears in the glass at the interface with oxide, which suggests a local composition gradient of the glassy phase.

After 15 min (Fig. 7(b)) the oxide scale has disappeared, giving way to a grey zone which belongs to the glass but with a different colour.

For longer times of 40 min it can be seen in Fig. 7 that crystallizations appear in the grey zone, which area is larger, with grey shades toward the glass. The thickness of the crystallizations is very variable, going from 2 to 10 μm .

Finally, for durations of 60 min (Fig. 7(d)) the glassy phase has reached again the alloy, there is no more crystallization and, in the alloy, pores and oxide pockets are scattered in a homogeneous way.

Fig. 8 presents the EDAX results obtained for cross-sections of the samples treated for 15 min (Fig. 8(a)) and 40 min (Fig. 8(b)).

At the beginning, we recognize from right to left in Fig. 8(a) the nominal alloy (part I), the Fe-depleted area, rich in cobalt and nickel (part II), then a thin zone with an high content in iron, containing silicon (part III) and, the last, the glassy phase (part IV). It is clear that significant amounts of iron have already diffused into the glass for times as short as 15 min, showing that there is a rapid reaction between the enamel and the superficial iron oxide. Nevertheless, no new crystalline phase is identified by X-rays analysis: so we can consider that it is a real iron oxide dissolution into the glass that occurs from the first few minutes. An analysis by atomic absorption of the glass confirms that there are some cobalt traces (about 1%) in the glass, but no nickel.

After 40 min the concentration profile of Fig. 8(b) presents a noticeably different shape. The thickness of part II, impoverished in iron is smaller, so confirming the progressive homogenization of the alloy. The same is seen in the glass, which also tends to homogeniza-

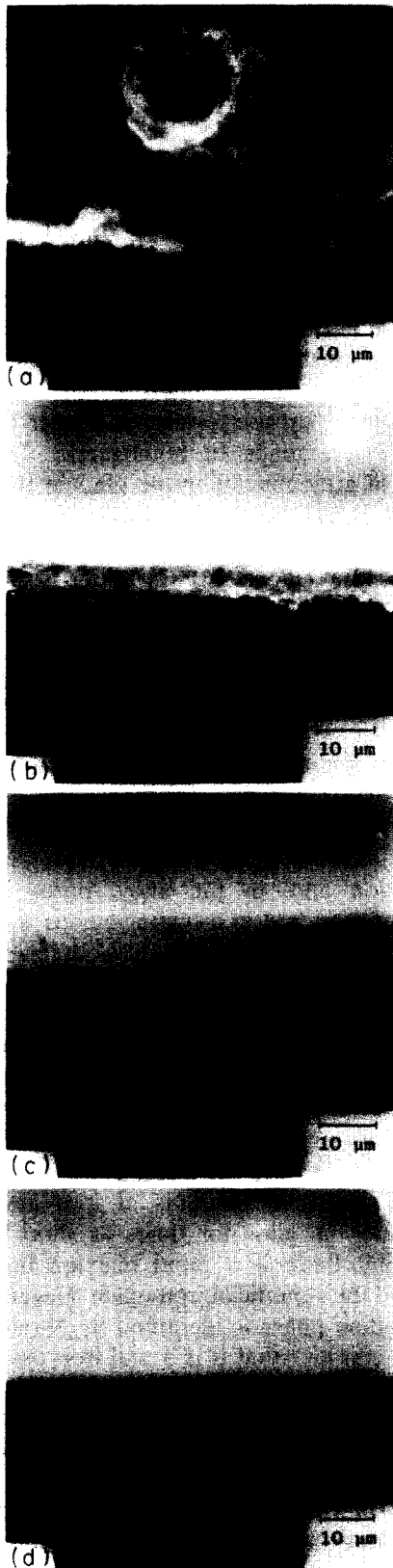


Fig. 7. Metallographs of the kovar–glass interfaces after (a) 3 min, (b) 15 min, (c) 40 min and (d) 60 min at 910 °C.

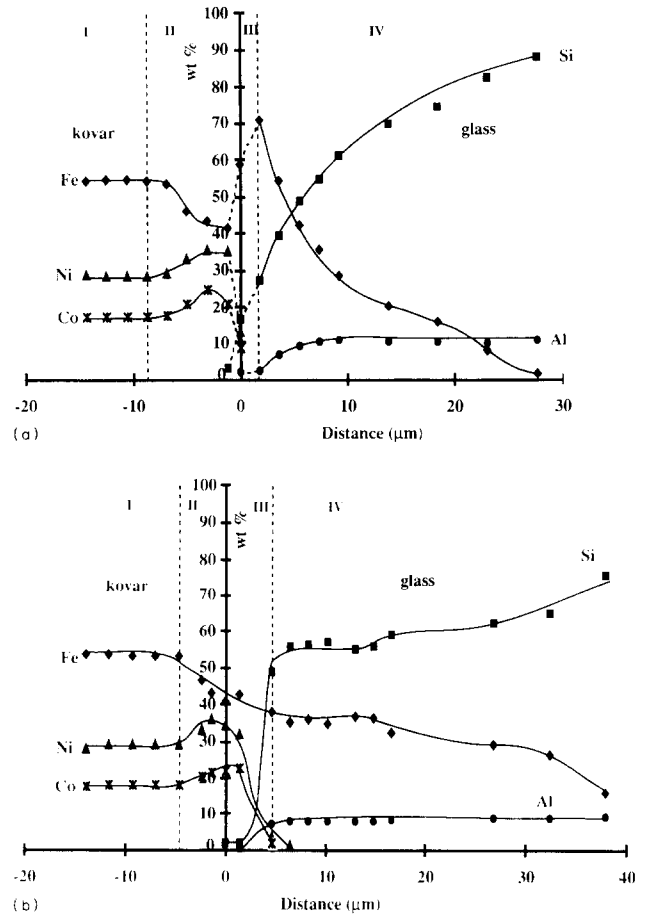


Fig. 8. Fe, Ni, Co, Si and Al concentration profiles in the interfacial zone after sealing at 910 °C for (a) 15 min and (b) 40 min.

tion: iron has diffused to more than 40 μm inside the glass when its maximum iron content does not exceed 38 wt.% instead of 70 wt.% after 15 min. In the interfacial zone III, with crystallites, there are glass and alloy components together. X-ray analysis identifies a crystallized phase isotypic with Fe_2SiO_4 with lattice parameters $a = 0.4799$ nm, $b = 1.0393$ nm, $c = 0.6063$ nm, smaller than those of fayalite ($a = 0.48215$ nm, $b = 1.04805$ nm, $c = 0.60902$ nm), maybe because of the presence of cobalt or nickel, or also of some element of the glass (aluminium, sodium or boron). Moreover, it is obvious that the boundary between parts II and III is not very clear, confirming that the glassy phase has really penetrated into the alloy.

Thus, during the thermal treatment, the oxide scale and the enamel react, forming first a glass rich in iron, which rapidly crystallizes in an Fe_2SiO_4 -like phase. At the same time, there is continuously a diffusion of iron into the glass, the ultimate result of this being the dissolution of the crystallized phase (which has disappeared after 60 min; cf. Fig. 7(d)). To explain better these two competitive steps, it is worth assuming that the iron (and perhaps a little cobalt and nickel)

diffusion enriches the glass up to a limiting content where a local devitrification may occur. However, this devitrification is not immediate, so that the glassy phase has time to dissolve the whole oxide scale

before crystallization. After this step, diffusion goes on but iron is then drawn from the previously formed crystalline phase, so involving its disappearance. The whole process does not last long, since the glass (and probably also the alloy) is completely homogenized after 60 min at 910 °C.

4.2. Influence of temperature

Temperature is taken up to 940 °C and soaking times go from 8 to 15 and 30 min. Looking at Fig. 9, it is clear that crystallization appears from 8 min (Fig. 9(a)) near the glass–kovar interface, taking the place of the oxide scale, with a very variable thickness of about 10 μm reaching sometimes a maximum of 18 μm. Crystallization seems to increase a little between 8 and 15 min (Fig. 9(b)), but after 30 min (Fig. 9(c)) the crystallites seem to be detached from the alloy and the glass has penetrated to the kovar.

X-ray diffraction identifies the crystalline zone always as a silicate isotypic with Fe_2SiO_4 .

Globally, the behaviour at 940 °C is very analogous to that observed at 910 °C, but crystallization begins more rapidly and with a greater extent. The dissolution of the crystallites also begins sooner, after 30 min or so, while it only occurs at about 40 min at 910 °C.

So, increasing temperature accelerates logically the two steps of the interfacial reaction, the crystallization as well as the iron diffusion, including the dissolution of the crystallized phase.

4.3. Conditions for a good sealing

Table 4 provides the results of sealing tests using different times and temperatures, and with kovar preoxidized for 3 and 5 min. The thermal treatment was always that described in Fig. 1.

The quality of the junction is assessed after three

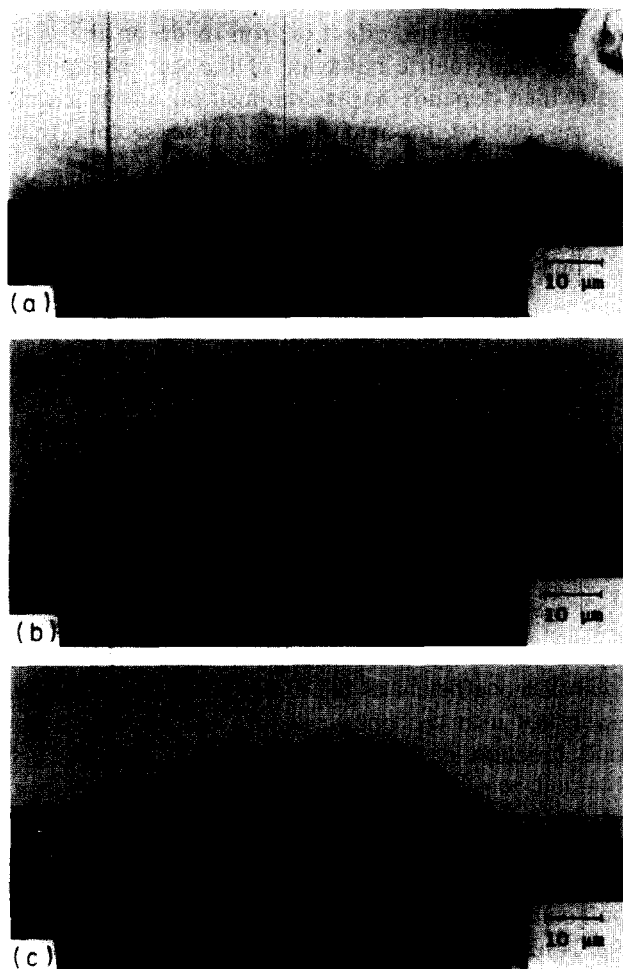


Fig. 9. Metallographs of the kovar–glass interfaces after (a) 8 min, (b) 15 min and (c) 30 min at 940 °C.

Table 4
Sealing conditions and results

Code of the test	Sealing temperature (°C)	Time of preoxidation (min)	Sealing time (min)	Crystallites at the interface	Oxide scale remaining at the interface	Quality of the junction
A	940	5	8	+	–	–
B			15	+	–	–
C			30	+	–	–
D	910	5	5	–	+	–
E			8	–	+	–
F			15	–	–	+
G			20	–	–	+
H			40	+	–	–
I			60	–	–	–
J	910	3	5	–	+	–
K			15	–	–	–

months and by an empirical test of the section of the samples for analysis. The symbol “–” is given for samples that became unstuck immediately after cooling, or after some weeks, or when the junction was destroyed by cutting with a diamond saw.

There is a clear relation between the presence of the crystallized zone and the poor quality of the junction (tests A, B, C and H). However, unsticking is also easy for samples without any crystallization in the glass, when sealing times are short (tests D, E and J), i.e. when the oxide scale is still remaining at the interface.

So, a good sealing may be only achieved when there is no crystallized extra phase (iron oxide or silicate) in the interfacial zone, between glass and kovar (tests F and G). However, this condition is not sufficient, because no bonding is obtained directly between non-preoxidized kovar and glass [2] and for too long sealing times (test I).

The necessity for the preoxidation of kovar has already been justified for its role in wetting [2] and, otherwise, the effect of too long sealing treatments (test I) has been shown [4] to create a maladaptation of the thermal expansion coefficient between the glass and the alloy near the interface, involving disastrous stresses during cooling.

Indeed, the preoxidation of kovar has three main roles [4]: first to form an oxide scale which favours the wetting, second to enrich the glass in iron by the dissolution of the oxide scale so that its thermal expansion coefficient reaches that of the interfacial alloy, and third to create a superficial open porosity in the alloy, allowing a penetration of the glass and hence a good physical adherence.

If preoxidation is not sufficient (test K), then no good bonding can be achieved even if the dissolution of the oxide scale is complete: the thermal expansion coefficient of the glass is not increased enough. So, a good sealing process requires that the following conditions are met.

(1) The kovar is sufficiently preoxidized. Too small oxide scales do not allow the glass to reach the dilatometric behaviour of the interfacial alloy; too thick oxide scales cannot be dissolved before crystallization in the glass.

(2) Temperature must be high enough to allow wetting, but not too high because crystallizations appear immediately.

(3) Time must be adjusted in such a way that oxide scale is entirely dissolved, but no crystallization has yet appeared.

For our materials the ideal conditions are 5 min of preoxidation of kovar at 700 °C, then enamelling porcelain following the cycle of Fig. 1 and sealing treatment with a soaking temperature of 910 °C for 15–20 min.

It is not possible to quantify, in a general way, the

optimal thickness of the oxide scale, as it depends, for sealing, on the following parameters:

- (1) the iron diffusion rate (varying with the nature of the glass and with the temperature);
- (2) the glass devitrification rate (for the same reasons);
- (3) the dilatometric effect of iron oxide on the glass;
- (4) the dilatometric behavior of the underlying alloy (which depends on its composition, itself depending on the preoxidation and the sealing treatment).

5. Conclusion

As expected, the difficulties in joining kovar to porcelain through a glassy interphase do not lie in the glass to porcelain interphase: if attention is paid to the fitness of dilatometric behaviour of glass and porcelain no cracks are observed. The problems are located at the glass to kovar interface with the dissolution of the oxide scale covering kovar after preoxidation.

At the beginning, a very rapid diffusion of iron into the glass leads to the complete dissolution of the oxide scale. If its thickness is appropriate, the quantity of iron dissolved gives to the glass the same thermal expansion coefficient as the underlying alloy. The glass penetrates into the open porosity of the alloy and a good junction is achieved. The “ideal interface” so obtained is constituted of iron-depleted kovar–iron-rich glass with neither dilatometric nor composition gap and without any crystalline ceramic phase. If there is not enough oxide, the fit of the thermal expansion coefficients is not good and the bonding fails. If the time is too long, then a significant devitrification of the glass occurs near the alloy interface; it is mechanically very poor and there is unsticking after cooling. Even if the time is long enough to ensure a complete dissolution of the crystallized silicate phase so formed, it is not satisfactory because the consecutive homogenization of the glass brings down its thermal expansion coefficient to values too weak to allow a good bonding.

So, the study of the mechanism of the reactions occurring during the sealing process makes it easier to understand why the conditions for obtaining good results are so precise and the industrial knowhow is so important.

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