A Study on the Solderability of Pure Nickel with different Tin-Lead Solders using Resin and Zinc Ammonium Chloride Fluxes

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Introduction

Nickel and its alloys can be successfully soldered using tin-lead solders[1]. For several engineering and electronic applications, either nickel and its alloys or other metallic materials coated with nickel are to be soldered. Even though the soldering practice for nickel has been in use for some years, data concerning systematic fundamental studies on the solderability of nickel with different solders and fluxes under different conditions of soldering are scarce in literature. In this work, an attempt has been made to study the effect of solder composition, nature of flux and soldering temperature on the efficiency of soldering and on the joint strength of butt and lap soldered joints of pure nickel.

Theoretical aspects

The efficiency of soldering[2] on a given base metal is measured in terms of the spreading coefficient, ϕ, given by the relation :

$$\phi = \gamma_{LF} (\cos \theta - 1) \quad \ldots \quad 1$$

Here, γ_{LF} is the solder-flux interfacial tension and θ is the contact angle of the solder under a cover of the flux on a base metal. When the solder drop is in a state of equilibrium,

$$\text{Cos } \theta = \frac{\gamma\mu_F - \gamma\mu_L}{\gamma_{LF}} \quad \ldots \quad 2$$

where γ_{LF}, $\gamma\mu_F$ and $\gamma\mu_L$ are the interfacial tensions between liquid solder-flux, metal-flux and metal-liquid solder respectively.

The interfacial tension, γ_{LF} is given by the relation :

$$\gamma_{LF} = \tfrac{1}{2}gh^2 (D-d) \quad \ldots \quad 3$$

Where D and d are density of solder and flux respectively and 'h'(3) is the height of the flat top of the solder drop from the equatorial girdle of the flat solder button resting on a horizontal plane.

From the physical point of view, complete spreading is characterized by a value of contact angle $\theta=0$, while complete non-wetting is given by $\theta=180°$. The spreading power ϕ in Eq. 1, therefore, takes a maximum value for $\theta=0$, and for all finite values of $\theta>0$, it is negative, reaching a minimum value of -2 (γ_{LF}) for $\theta=180°$. Thus, while $\phi=0$ indicates complete spreading, all other values indicate different degrees of non-wetting. It is also seen that ϕ depends on the liquid solder-flux interfacial tension, γ_{LF}, and in this way the spreading efficiency improves if γ_{LF} is lowered or $\gamma\mu_L$.

Materials and Experimental procedure

Solders : Analar grade tin, lead were used in the preparation of the alloys. The constituent metals were weighed and melted together under a cover of resin flux.

Base plate : 2.5 cm $\times$ 2.5 cm size base plates were cut from 99.9% nickel, 2.5 mm sheet, ground on emery

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papers of successively finer grit up to 4/0, cleaned, degreased with Trilene and stored in a desiccator for contact angle measurements.

Fluxes : Resin and zinc ammonium chloride were selected as representatives of the 'protective' and 'reactive' type of fluxes respectively[4]. Resin flux used was of commercial quality, melting point 108°—111°C, density 1.07 gms/cc. The zinc-ammonium chloride flux was prepared by melting a mixture of 79 parts by wt of zinc chloride and 21 parts of ammonium chloride (A. R. Quality) and stored in a desiccator[5], melting point 179°C, density 2.3 gms/cc.

Methods : The solder-flux interfacial tension was determined, employing the method of Chalmers and Wadie[3] and the values are reported earlier[6] for pure metals and nine intermediate compositions. Contact angle measurements were made by an optical method [3,7]. The spreading coefficients[2] have been computed by employing Eq. 1.

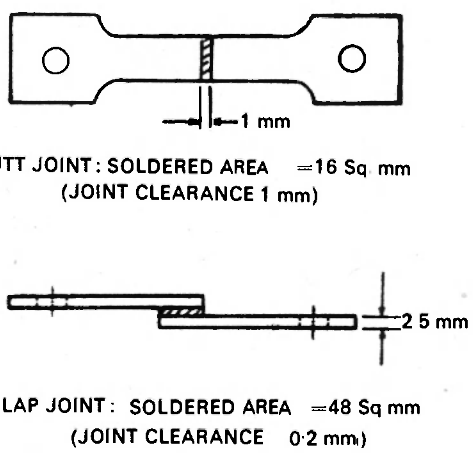


Fig. 1. Schematic diagram of lap and butt joint assemblies.

For making butt soldered joints, sheet specimens of dimensions shown in Fig. 1 were prepared and the soldering end faces were ground and cleaned as before and were held in a graphite jig. This assembly was placed in a muffle furnace and uniformly heated to a temperature of 30°C above the respective liquidus temperature of the solders, maintained for 20 minutes and allowed to cool in the furnace itself. Lap joints (Fig. 1) were also prepared in a similar fashion employing a graphite jig having an appropriate step in the middle. The soldered specimens were then tested in a Hounsfield tensometer fitted with a 250 Kg spring beam at a constant strain rate of 1.5 mm/min at room temperature. Lap joint strength was determined by using special grips to facilitate proper deformation of the solder in the tensile direction[8,9,10].

Area of spread was measured using a Aristo planimeter. For this purpose, the base plate with solder and flux was heated to 300°C and maintained for 20 minutes and allowed to cool in the contact angle furnace itself.

Results and Discussion

Contact angle : The contact angle of solders on pure nickel, using resin and zinc ammonium chloride fluxes were measured at their respective liquidus temperatures and upto a temperature of 300°C are presented in Table I. The contact angles at the respective liquidus temperatures of solders are plotted in Fig. 2 for both the fluxes. It can be noted that the contact angle value for a given solder and flux decreases with increase of temperature. In general, contact angle values with resin flux are greater than those obtained with the chloride flux and this can easily be attributed to the reactive nature of the chloride flux. A reference to Fig. 2 indicates that the contact angle values for the two pure metals are high compared to those for the tin-lead alloys with the chloride flux. The values indicated in Table I show that the contact angles drastically decrease to very low value with increase of temperature for the

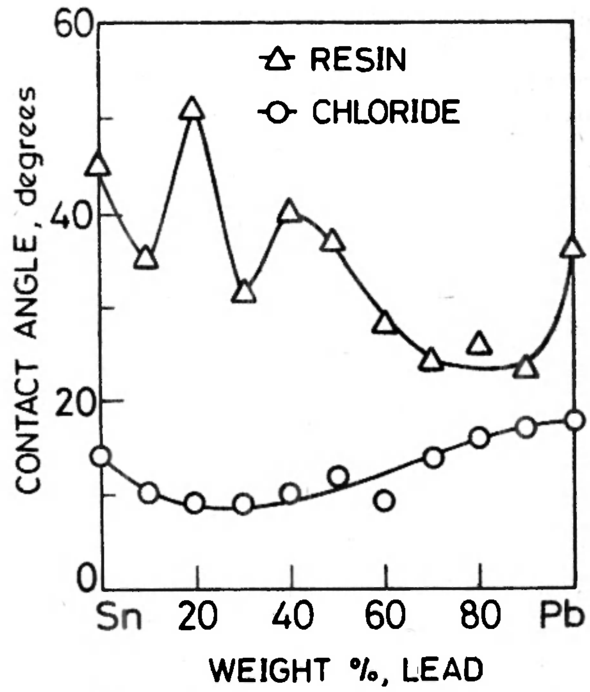


Fig. 2. Variation of contact angle at the liquidus temperatures of solders with composition.

Table I : Contact angle of Tin-lead solders on pure Nickel under Resin and Zinc ammonium chloride fluxes

Solder Composition Tin — Lead % %	Liquidus temp.°C	Contact angle (degrees) Resin flux Liquidus	260°C	280°C	300°C	Zn-NH_4Cl flux Liquidus	260°C	280°C	300°C
100— 0	232	45	36	29	28	14	13	8	7
90— 10	225	35	26	24	20	10	6	5	<5
80— 20	205	51	41	29	19	9	5	<5	<5
70— 30	190	32	15	11	8	9	<5	<5	<5
60— 40	185	40	17	9	7	10	<5	<5	<5
50— 50	220	37	18	11	9	12	<5	<5	<5
40— 60	235	28	11	9	8	9	<5	<5	<5
30— 70	260	24	—	16	15	14	—	<5	<5
20— 80	278	26	—	—	18	16	—	—	>5
10— 90	300	23	—	—	—	17	—	—	—
0—100	327	36	—	—	—	18	—	—	—

chloride flux. With resin flux, contact angle values are high for tin, lead and 80:20 and 60:40 tin-lead alloys. It is interesting to note that a similar trend has been observed for the variation of contact angle of different tin-lead solders on pure copper[11]. In tin-lead solders, tin is an active constituent, promoting wetting of the base metal and lead acts as a diluent affecting the viscocity and surface tension of the solder[12]. The factors responsible for the variation in contact angle are (i) the change in the liquidus temperature caused by the addition of lead, (ii) the variation in the extent of alloying between the solder and the base metal and (iii) the efficiency of flux at the temperature of soldering. The specific shape of the contact angle composition curve for resin flux (Fig. 2) is a result of the combined effect of all these factors.

Formation of halo's

The formation of halo's has been discussed by Bailey and Watkins[12] and Turkdogan and Zador[13]. Bailey and Walkins[12] stated that the 'halo' of intermetallic compound was visible ahead of the advancing pool of molten solder. They also showed that, lateral diffusion of liquid metal atoms into the substrate at the advancing periphery of liquid controls the rate of spreading of the solder. Turkdogan and Zador[13] observed that there is a noticeable increase in the area of spread for the spreading of tin on mild steel when the zinc chloride based flux was used, and this was not noticed when hydrogen atmosphere was employed in place of the chloride flux.

Fig. 3 gives macrographs of solder drops on nickel substrate employing the zinc ammonium chloride flux for 70, 60 and 50 per cent tin solders heated up to 300°C for 20 minutes. It can be noticed that the width of the band decreases with decrease of tin content in the solder. It was further observed that halo formation was not evident for other solders for the chloride flux and for all the solders for the resin flux and these findings are in agreement with other works [12,13].

Area of spread

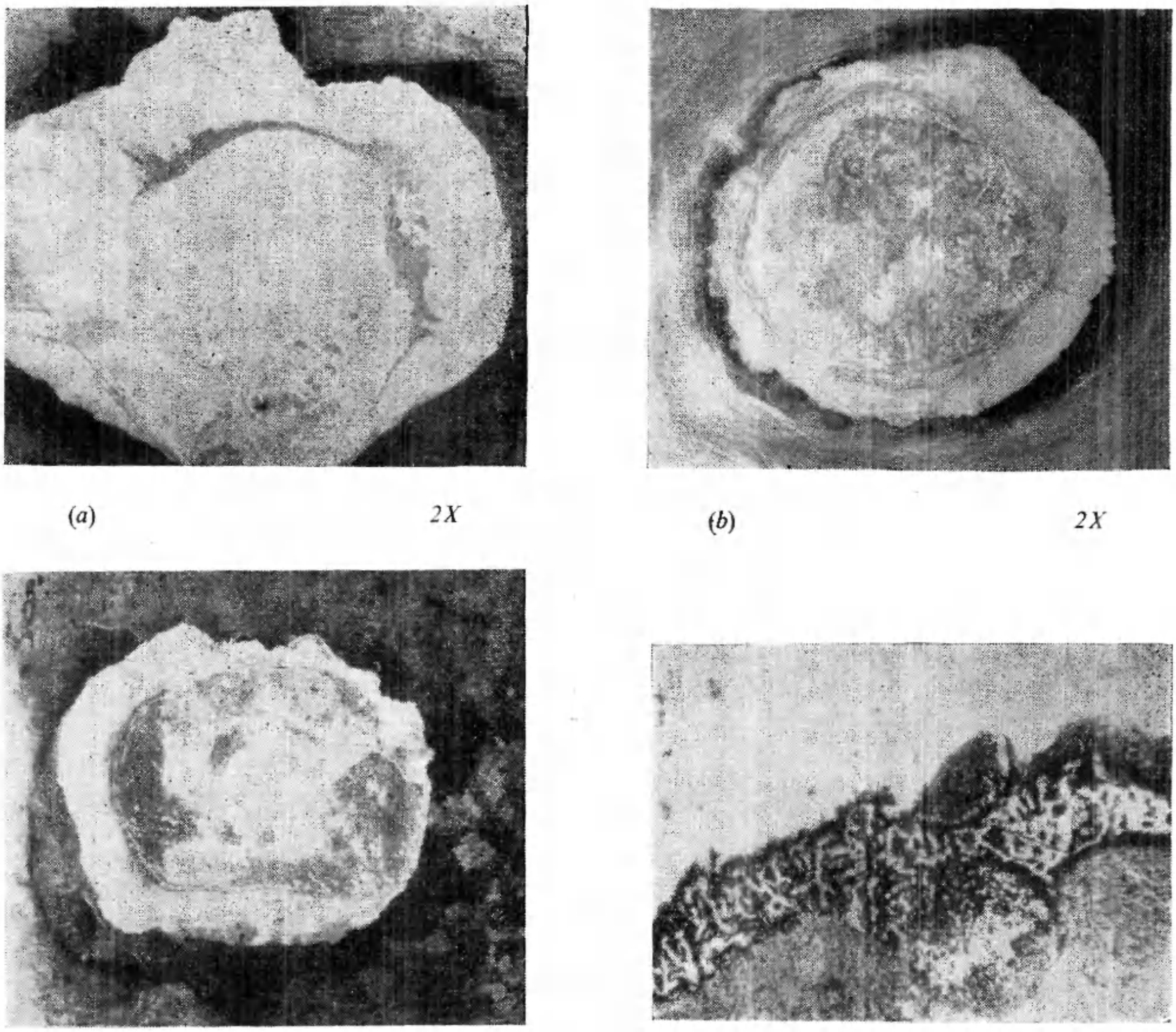
Fig. 4 shows a plot of area of spread vs. composition of the solder for both the fluxes. The area of spread for chloride flux is higher than that for the resin flux. The band that forms around the solder (Fig. 3) has not been taken into account in measuring the area of spread. Maximum area of spread occurs around the eutectic composition on either side of which the area of spread decreases steeply. This observation is in good agreement with earlier findings[14,12].

Spreading coefficient

The variation of spreading power of tin on pure nickel with progressive addition of lead is shown in Fig. 5 at 280° and 300°C for resin and zinc-ammonium chloride fluxes. It is observed that spreading coefficient values with tin, high-tin alloys and lead-rich alloys are much smaller when compared with those for intermediate alloys of tin and lead for both the fluxes. The spreading coefficient values with the chloride flux are greater than those with the resin flux. It is interesting to note that the spreading coefficient values for alloys in the range of 70 to 30 per cent tin are high for both the fluxes and at the highest temperature of soldering 300°C, resin flux appears to be as effective as the chloride flux. Of all the tin rich alloys studied with resin as flux, 80:20 tin-lead solder has very low spreading coefficient value at 280°C. This finding is in good agreement with the results obtained on commercial brass[6] and mild steel[15].

Strength measurements

Tensile strength of butt joints and shear strength of lap joints were measured and plotted in Fig. 6a, 6b respectively for both the fluxes. A reference to Fig. 6a indicates that 40:60 tin-lead solder exhibits maximum strength and this decreases on either side with the addition of tin or lead. However, alloys with 70 to 30 per cent tin exhibit good strength and strength values are almost in the same range with both the fluxes. Shear strength



(a) 2X *(b)* 2X

(c) 2X *(d)* 2X

Fig. 3. Macrographs showing band or 'Halos' formed on nickel surface surtounding molten solder of (a) 70 : 30 Sn-Pb, (b) 60 : 40 Sn-Pb and (c) 50 : 5 Sn-Pb solders with the chloride flux; (d) Magnified view of band of 60 : 40 Sn-Pb solder with the chloride flux on nickel.

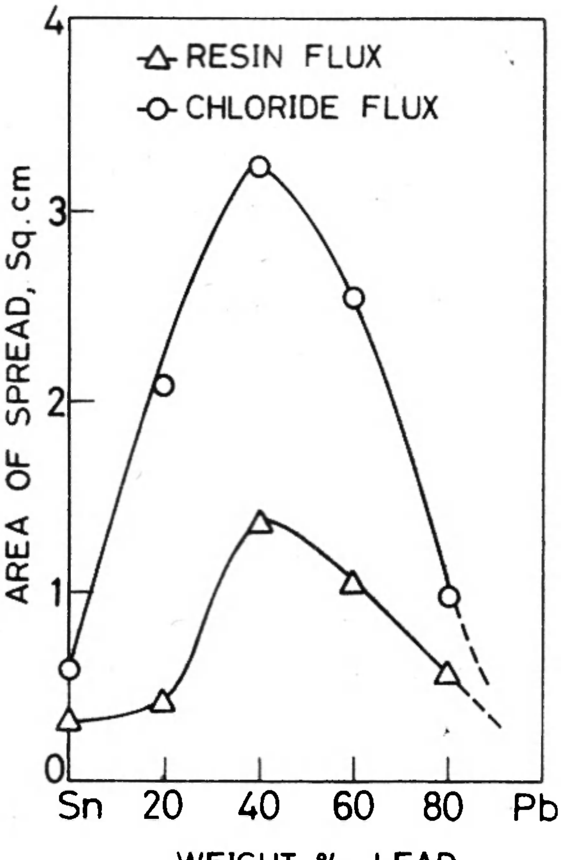


Fig. 4. Area of spread versus composition of the solder.

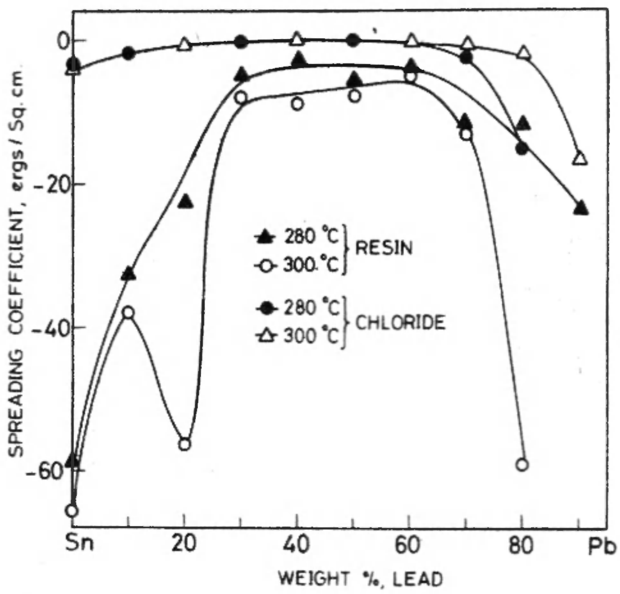


Fig. 5. Effect af addition of lead on the spreading power of Tin.

values (Fig. 6b) for resin show that 80:20 tin-lead solder exhibits poor strength and the 60:40 tin-lead alloy exhibits the highest strength. This sort of variation was observed in copper, brass and mild steel butt soldered joints[16] with the chloride flux. Maximum strength was observed at 60 per cent tin and further addition of tin or lead only decreased the strength value. Similar results were obtained for variation of shear strength of copper soldered joints made with different tin-lead solders by Nightingale[4]. Formation of soldered joints is a complex phenomenon and the strength of joint is influenced by the type and extent of intermetallic compound that may be formed. Fig. 7 shows a photomicrograph of an interface between nickel and 60:40 tin-lead solder exhibiting intermetallic compound formation. Considering both fluxes and the two types of joints, it is observed that between 40 to 60 per cent tin is favourable for formation of stronger joints.

Summary

Thwaites, in his excellent review article[17] has discussed the complexities involved in studying and quantitatively assessing the efficiencies of different soldered joints and the important parameters involved there-in. He has come to the conclusion that the majority of commercial soldering difficulties do not arise from the soldering technique or the fluxes and solders, when they are scientifically selected. Table II gives the performance characteristics of different tin-lead solders for soldering nickel. It is clear that intermediate tin-lead compositions are superior to tin as well as the high tin-lead alloys. It is also evident that whenever the spreading characteristics are good, the mechanical strength of joint will also be high. This sort of relationship has been reported earlier for copper, commercial brass and mild steel joints made with different tin-lead solders with both the fluxes[16]. In general, zinc ammonium chloride flux appears to be more effective than the resin flux but where corrosion action of the chloride flux cannot be tolerated, resin flux can also be effectively used. Earle[14] has demonstrated that the eutectic tin-lead solder can be as good a solder as a best solder can be and the above results show that nickel soldering is no exception to this.

Acknowledgement

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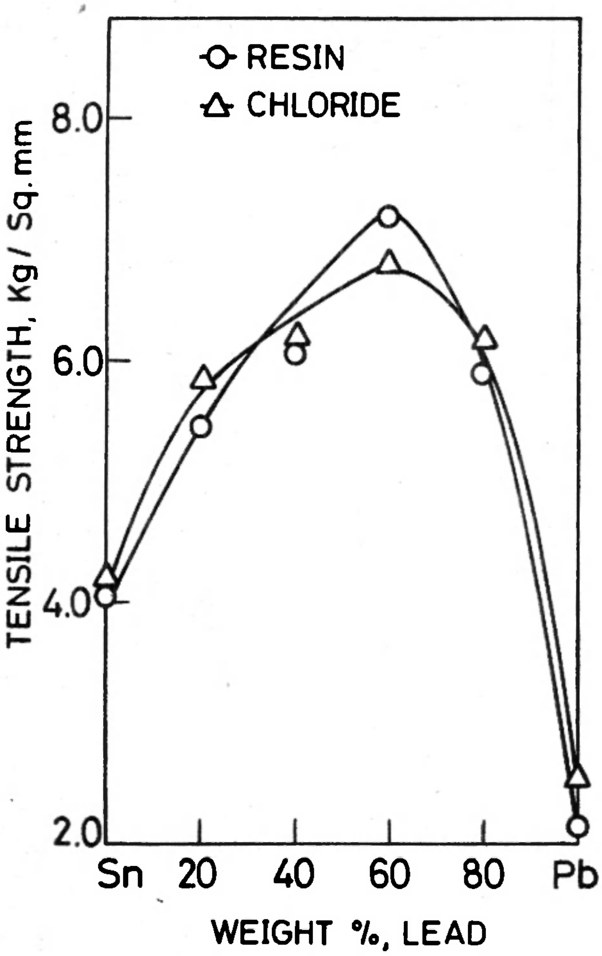

Fig. 6a. Effect of composition on the tensile strength of butt soldered nickel joints.

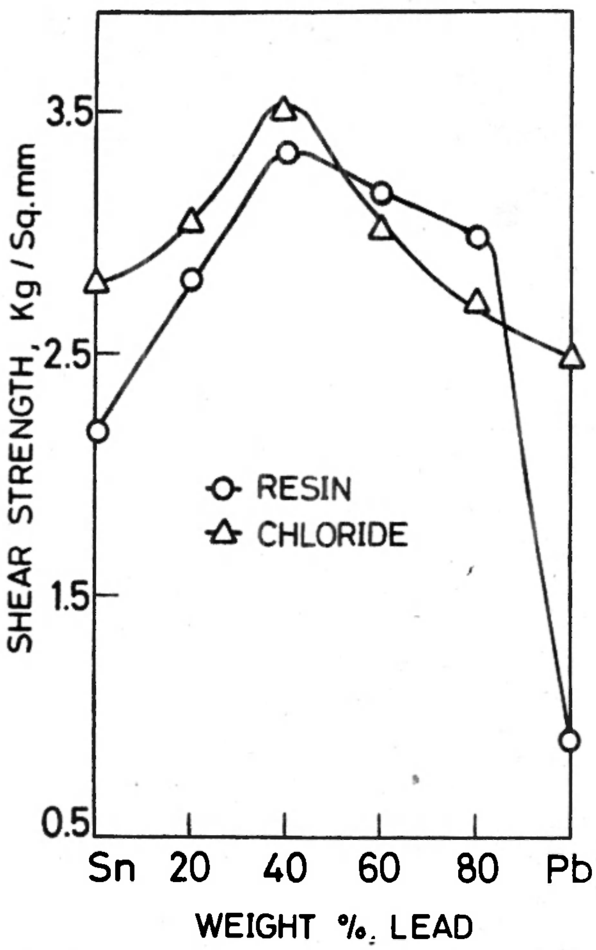

Fig. 6b. Effect of composition on the shear strength of laps soldered nickel joints.

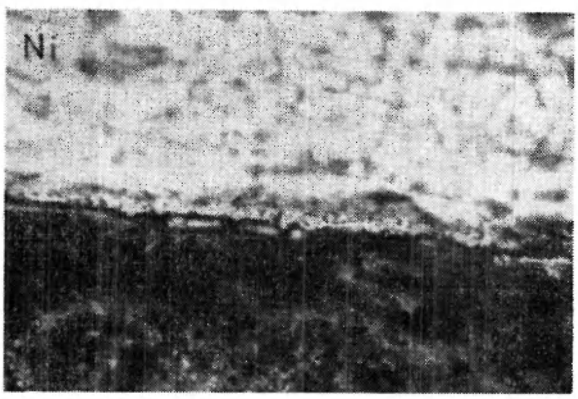

Fig. 7. Photo micrograph of the interface between nickel and 60 : 40 Sn-Pb solder 600X,

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Table II : Performance characteristics of Tin-Lead solders on Nickel

Solders Tin—Lead % %	Liquidus temp.°C	Spreading coefficient (300°C) A	B	Area of spread, (300°C) A	B	Tensile strength A	B	Shear strength A	B	Remarks
Tin	232	V. Low	High	Poor	Poor	Ave.	Ave.	Good	Ave.	This solder is not useful for high joint strength and resin cannot be a good flux.
80—20	205	Low	V.High	Poor	Good	Good	Good	Ave.	Good	An average solder with both the fluxes.
60—40	185	High	V.High	Ave.	Excel.	Good	Good	Excel.	Excel.	Very good solder with high joint strength, can be used effectively with both fluxes for low temp. soldering application.
40—60	235	High	V.High	Ave.	Good	Excel.	Good	Good	Good	Solder having good spreading characteristics with both the fluxes.
20—80	278	Ave.	High	Poor	Poor	Good	Good	Good	Good	This solder can be used effectively for high temperature soldering having comparatively good strength.

A =Commercial Resin flux, B=Zinc-ammonium chloride flux.

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