Solder Alloy Development for Automotive Radiators

BY R. E. BEAL

Tensile-peel tests on Pb-Sn-Ag solder alloys indicate properties superior to conventional Pb-Sn solder alloys for radiator applications.

ABSTRACT. Automotive radiator operating temperatures and pressures have gradually increased over the years, restricting the margin between service duty conditions and the capabilities of present systems. Joint stresses in service impose a tensile-peel load on the radiator soldered joint, but soldered joint mechanical properties are not usually expressed in this manner. This study examines the mechanical properties of conventional lead-tin solders and one lead-tin-silver alloy by the tensile-peel method, revealing that the silver-bearing alloy has much superior strength properties. Experimental solders in the high lead region of the lead-tin-silver system are then investigated, and new compositions are proposed based on the higher strength properties measured in these solders.

Introduction

The automotive radiator was initially conceived as a soldered brass and copper product subject to low and easily accommodated temperatures and pressures. Over the years, engine design has required a gradual increase in both parameters. The soldered joint has changed its role, consequently, from a mere water seal to a structural member of the system. Current solders are adequate at the present time, but the margin between operating parameters and radiator capabilities has markedly narrowed.

Future performance demands improved solders and soldering techniques if the present method of construction is to be retained. Special concern with quality control and product uniformity must coincide with the use of new materials so that a cost-effective satisfactory radiator can be made.

Fig. 1—Solder-joint strength as a function of alloy composition

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has in the past encompassed basic solder—base metal reactions, an understanding of soldering mechanisms, and process studies. \(^1\) The information given in this paper is aimed at detailing meaningful engineering properties of existing solders and presenting development of new solder alloys to meet the strength requirements of imminent temperature and pressure increases in the automotive radiator.

**Background on Solder Joint Mechanical Property Determination**

Mechanical properties of soldered joints are related to the test method used to derive them. Careful selection of the test method to reflect the loading characteristics in service is necessary if the data are to be useful in practice. The automotive radiator tank is essentially a pressure vessel, and examination of the tank joint reveals that it is subjected to tearing forces or a tensile-peel action when in operation.

Most of the mechanical property data available on the soldered joint, however, have been measured using a lap-shear specimen. Work by Nightingale and Hudson \(^5\) showed the variation of shear strength of soldered joints as a function of composition across the entire lead-tin system. Maximum strength values were found at approximately 50% tin content in joints and at 60% tin in bulk solder specimens, as in Fig. 1. Interestingly, the mechanical properties of the various lead-tin alloys tested under tensile-peel conditions do not appear to follow the same pattern. \(^3\) Room-temperature properties of high lead alloys were better than the eutectic lead-tin alloy in a tensile-peel test. Thus, the test method directly affects the results and the rank order of solder alloys. Loading characteristics of joints in service, therefore, must be carefully assessed for appropriate test method selection.

An explanation for some of the differences in mechanical properties between lap-shear and tensile-peel may be found in Fig. 2, which presents more of Nightingale’s work. Here, a definite relationship between soldering temperature and joint thickness (joint clearance) for maximum joint strength with all lead-tin solders in lapped specimens was established. Superimposed on the graph are solder alloys and soldering temperatures at which optimum strengths were achieved using the tensile-peel specimen. The optimum soldering temperature to give maximum strength properties for the 50% tin alloy with a tensile-peel specimen coincides with the predicted soldering temperature for maximum lap joint strength at a joint clearance of 0.004 in., which happens to be the physical optimum clearance for lap-shear specimens. The relationship appears to be more than just coincidental. Optimum tensile-peel strength soldering conditions for the 30% tin alloy and the 15% tin alloy are also presented for comparison. Clearly, at high temperatures narrow clearances are necessary to prevent solder run-out, and with wide clearances a relatively sluggish low temperature solder is preferred to fill the joint. In addition, with less than 0.004 in. clearance, flux inclusions and void formations tend to occur and reduce strength properties. Also, solder tends to run out at the high solder temperatures required for good strength properties in high lead alloys. These purely physical considerations dictate that a clearance of approximately 0.004 in. is the optimum for the lap-shear joint, and only one lead-tin solder alloy with 50% tin is at its optimum soldering temperature at this clearance.

Work by Chadwick \(^6\) was the first attempt to test a soldered joint under tensile-peel conditions, although he restricted himself to the reporting of steady-state load or propagation load data. Expansion of his technique to include fracture initiation informa-
tion and an entirely different specimen preparation procedure forms the basis of the mechanical property determinations in this paper. Previous work determined that the static room-temperature strength of soldered joints depended on the solder alloy, the temperature and time of liquid solder contact, the specimen, and the testing method. Also, the soldering flux used can dramatically affect the data obtained. Zinc ammonium chloride flux gave the best results and was used throughout the program.

Experimental Procedure

Many factors influence the mechanical properties obtained from a soldered joint. For example, the single-lap joint should act in perfect shear but rarely does, because a bending moment is produced by joint asymmetry and introduces a certain amount of tearing action. This is somewhat overcome in the double-lap joint. The tensile-peel specimen defines two important features of the soldered joint — that is, the load to initiate a fracture and the load to propagate that fracture. Experience with the test has shown that results achieved are compositionally sensitive.

Samples, 7½ in. long by 1½ in. wide, of 0.005-in. strip of Alloy 260 were slightly abraded to mechanically clean the as-received surface and were bent into a U-shape for exposure in the solder. The ⅛-in. space between the two sides of the U-bend allowed for adequate solder flow between the two legs of the specimen and precluded base metal dilution effects on the solder in the joint area. Quadruplicate specimens for each test condition were exposed for 5 sec in 30 g of fresh solder heated in a glass container within a stainless steel beaker as shown in Fig. 3. By inserting a water-cooled copper heat sink after the 5 sec exposure, fast cooling rates were achieved.

Mechanical tests were performed on the specimens using an Instron tensile testing machine at a strain rate of 0.1 in./min. The ends of the legs of the U-bend specimen were held, and a tensile peel result was obtained. Two load values were recorded: that required for initiation of fracture and that required for subsequent fracture propagation. A typical load-extension curve and the method of loading are seen in Fig. 4.

Conventional Solder Studies

Mechanical Properties

Four conventionally used alloys were selected for the evaluation: 70 Pb-30 Sn; 85 Pb-15 Sn; 37 Pb-63 Sn; 97.5 Pb-1.0 Sn-1.5 Ag.

Soldering parameters for each alloy were selected from the previous work. Conditions used were 850°F exposure for 5 sec at temperature for the 97.5 Pb-1.0 Sn-1.5 Ag alloy and the 85 Pb-15 Sn alloy, 680°F exposure for 5 sec at temperature for the 70 Pb-30 Sn alloy, and 500°F for 5 sec for the 37 Pb-63 Sn alloy. An argon gas atmosphere was used during specimen soldering to maintain a dross-free solder surface. Mechanical tests were performed at room temperature and four
Elevated temperatures (150, 200, 250, and 300°F).

Elevated temperature testing was carried out in heated air in a heat-resistant glass container supplied with air from a fan heater. Preliminary tests were executed on dummy specimens with inserted thermocouples to determine the time necessary for temperature equalization of the specimen. Temperature was controlled by a thermostat on the fan device, and a mercury-in-glass thermometer indicated the temperature obtained. The setup is illustrated in Fig. 5. Glass wool insulation was placed at the jaws of the tensile machine for protection and retention of the heated air. The test procedure gave very satisfactory and consistent results.

The elevated temperature mechanical tests performed are summarized in Figs. 6 and 7. The silver-bearing alloy gave consistently better results throughout the temperature range as measured by fracture initiation loads, varying from over 90 lb per in. average at 300°F. The 70Pb-30Sn, 85Pb-15Sn, and 37Pb-63Sn alloys gave decreasing strengths with increasing test temperatures. Fracture propagation data showed that the high tin alloy had the highest strength at room temperature and lowest at 300°F. All alloys ranked in the same order at 300°F using either fracture initiation or propagation values.

The superiority of the silver-bearing alloy as measured by a static elevated-temperature tensile-peel test was very clear and suggested that a new exploration of the lead-tin-silver system — incorporating the soldering processing criteria established in prior programs and the tensile-peel mechanical test specimen — may reveal better alloys.

**Joint Fractures**

Specimen examination by the scanning electron microscope was particularly revealing. An 85Pb-15Sn solder joint with good properties is shown in Fig. 8. Very fine ductile dimples are present, and the joint gave good mechanical strength. The 85Pb-15Sn alloy also has a tendency to hot tear during solidification. A sample of hot tears is shown as Fig. 9. An area of fracture parallel to the major dendrite growth direction is shown. Failure has occurred by multiple shear of the secondary dendrite arms with an obviously low percentage fracture area. Several dendrites with considerable interdendritic shrinkage and very little interconnection can be seen.

Even though the laboratory techniques used for sample preparation are consistent, variations still occur in strength properties of good joints. One answer can be found in the fracture initiation point. Figure 10 shows a ductile dimple with its fracture initiation area clearly defined. Because of the lack of coherence between solder in bulk and the initial material solidified, a small area of the joint can separate easily and become the initiation point for fracture. The incidence and severity of these areas will influence the strength properties that can be measured on any given joint.

It may be that these variations will, by the very nature of the soldering process, always be present to some extent. Increased knowledge of the soldering action by fracture studies has proved to be valuable in improvement of soldering techniques.
New Solder Alloy Development

Alloy Selection

The high lead corner of the lead-tin-silver system was examined for possible new solders. Constitutional information was basic to the investigation. A detailed lead-tin-silver ternary diagram was not readily available to assist in alloy selection; however, a compilation of the liquidus surfaces in the system by Earle was located. Additional data on the respective binary systems are available in Hansen. A eutectic trough traverses the diagram from 97.5% Pb on the lead-silver side, proceeding to approximately 1.4% Ag, 3% Sn, remainder lead, and almost parallel to the tin side forming a ternary eutectic with Ag$_3$Sn at 62.5% Sn, 36.15% Pb, and 1.35% Ag at 352°F, according to Parravano. Tin solubility in lead is 1.9 wt% at room temperature and silver solubility is almost negligible.

The addition of alloying elements tends to increase strengths achieved. Solid solution strengthening is generally the best method: precipitation of a second phase or dispersion is useful if agglomeration or overaging can be prevented. Materials of face-centered cubic structure such as silver have relatively steep stress-strain curves, whereas both tin and lead have flat curves; thus silver should provide a degree of strain hardening and strengthening. The unit strengthening effect of a metal entering solid solution varies inversely with its solid solubility and directly with the difference in size of the solvent and solute atoms or the change in lattice parameter. In heterogeneous alloys, the strengthening depends upon the quantity and distribution of the new or second phase.

Creep, recovery, and recrystallization are also important features to be considered in evaluating metals for elevated temperature service. Recrystallization studies carried out some years ago at IITRI on lead-base alloys showed that nearly all binary alloys have higher softening temperatures than the base lead material. Solid solution strengthening was limited to some extent, and strengths generally quickly decreased above room temperature. Both silver and tellurium additions appeared to give alloys particularly resistant to high-temperature softening. The effect of silver on lead-tin alloys is to increase the recrystallization temperature, creep resistance, and resistance to deformation. Silver also increases measured hardness at elevated temperatures.

Other investigators have examined the lead-tin-silver system for better solder alloys. Present studies differ in two ways. Firstly, the tensile-peel test specimen with close soldering parameter control is used and, secondly, deliberately small increments of tin and silver additions are made. The compositional sensitivity of the tensile-peel test makes this approach possible. Alloys in the lead-rich corner of the ternary system comprise solid solutions and two-phase alloys of tin and lead, possibly with Ag$_3$Sn intermetallic dispersion as a strengthening medium. Solder alloys with tin contents up to 6% and silver additions up to 2% were studied to determined the maximum strengthening effects in this region of the system.

Fig. 11—Influence of alloy composition on fracture initiation strength properties

Fig. 12—Influence of alloy composition on fracture propagation strength properties

Fig. 13—Fracture initiation strength (lb per in.) of lead-tin-silver alloys soldered to alloy 260 brass at 850°F for 5 sec
Table 1—Mechanical Properties of Soldered Joints Tested at Room Temperature

<table>
<thead>
<tr>
<th>No.</th>
<th>Alloy Composition</th>
<th>Solder temp. °F</th>
<th>Fracture initiation strength lb per in.</th>
<th>Fracture propagation strength lb per in.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Series</td>
<td>Avg</td>
</tr>
<tr>
<td>7</td>
<td>95 Pb-3.8 Sn-1.2 Ag</td>
<td>680</td>
<td>124.0</td>
<td>31.1</td>
</tr>
<tr>
<td>9</td>
<td>69.9 Pb-29.6 Sn-0.5 Ag</td>
<td>680</td>
<td>118.0</td>
<td>31.0</td>
</tr>
<tr>
<td>7</td>
<td>95 Pb-3.8 Sn-1.2 Ag</td>
<td>850</td>
<td>64.4</td>
<td>24.3</td>
</tr>
<tr>
<td>9</td>
<td>69.9 Pb-29.6 Sn-0.5 Ag</td>
<td>850</td>
<td>49.6</td>
<td>25.6</td>
</tr>
</tbody>
</table>

Results
A preliminary series of experiments was performed with two selected lead-tin-silver alloys to establish preferred soldering temperature.

A high lead-tin-silver composition of 95Pb-3.8Sn-1.2Ag was selected and a higher tin content alloy, 70Pb-29.6Sn-0.5Ag, chosen for comparison. These alloys were soldered at 680 and 850°F for 5 sec at temperature, and mechanical tests by tensile-peel were executed at room temperature. Results obtained are presented in Table 1. The high lead-tin-silver alloy produced substantially higher strength property data than the high tin alloy. A soldering temperature of 850°F gave superior results with a fracture initiation strength average of 136.3 lb per lineal in., and was selected as the soldering temperature to be used for other experimental compositions.

The results were very promising in that substantial strength increases over the widely used 97.5 Pb-1.0 Sn-1.5 Ag alloy was achieved.

Other alloys, within the range 1-6% Sn and 0-2% Ag, were selected and manufactured from virgin materials to determine their mechanical strength properties at room temperature. Quadruplicate specimens were made at 850°F, and the property data presented are the averages obtained.

Several of the selected compositions produced tensile-peel strength properties that were superior to presently used alloys, as shown in Fig. 11. Maximum fracture initiation strength was achieved at 3.0% Sn-1.5% Ag, with strength gradually decreasing above this tin level. Alloys were not made between 1 and 3% Sn during the program, and some additional strength may be found in this region. Silver additions caused more complex behavior. Strength properties decreased with silver additions up to 0.8%, followed by an increase in strength with further additions up to 1.5%. Above this figure, strength properties again appear to diminish. Fracture propagation strengths increase with higher tin contents and are gradually reduced with increases in silver content as seen in Fig. 12.

Fracture initiation data have been plotted on the ternary diagram in Fig. 13. The eutectic trough is added to constitutionally relate the property variations measured with the experimental solders. Alloy strength properties are seen to vary rapidly in the eutectic region, and further exploration could be worthwhile.

Studies have revealed lead-tin-silver solder alloys with superior tensile-peel strength properties over the commercial composition. A 45% strength increase was registered with the 3.0% Sn-1.5% Ag solder. Good strength results occur with 3-4% Sn and 1.2-1.5% Ag which may enable manufacturers to improve the pressure capabilities of the radiator.

Radiator operating temperatures will also be raised, which makes the examination of soldered joint properties at elevated temperatures mandatory to determine whether the observed strength gains are retained. A total of twenty specimens of the new Pb-3.8Sn-1.2Ag alloy were made and subjected to room temperature and elevated temperature mechanical tests. Test temperature was raised at 50°F intervals. The results are compared with previous data generated with the commercial 97.5 Pb-1.0Sn-1.5Ag solder in Figs. 14 and 15. Fracture initiation strength data show a definite improvement in properties both at room and elevated temperatures, with 30% and up to 100% strength increases, respectively. The extra tin content of the new composition also assists in raising the fracture propagation strength.

![Fig. 14—Comparison of elevated temperature initiation strength properties](image1)

![Fig. 15—Comparison of elevated temperature fracture propagation strength properties](image2)
measured in Fig. 15. Large gains in high temperature strength as measured by the tensile-peel method have been made. All tests were pulled at 0.1 in./min strain-rate within 5 min of reaching the specified test temperature. Further work is necessary on the dynamic properties of the new solder alloys, preferably under simulated radiator operating conditions before present solders can be replaced. The properties measured are very encouraging and should lead to new commercial solder compositions.

Main implications of the work for radiators are:
1. The need to closely control solder alloy composition and processing parameters for maximum utilization of inherent solder strength.
2. The possibility of new compositions in the lead-tin-silver system for improved joint strength properties.
3. Further exploration of new solder alloys and generation of dynamic test data would be beneficial.

Surface and Fracture Examination of Modified Solder Alloys

Because of differences in constraining forces, surface crystal behavior is not necessarily the same as internal grain behavior. Nevertheless, valuable information can be derived from examination of the surfaces of experimental alloys after mechanical testing is performed. Six of the modified alloys were examined on the fracture interfaces and bulk solder surfaces adjacent to fractures.

Grain boundary influence is markedly dependent upon strain rate and temperature. All the samples examined here were strained at 0.1 in./min at room temperature, and the remarks made should be viewed correspondingly. For future work, elevated temperature testing at various strain rates would add more knowledge on the kinetics of deformation in lead-rich alloys. Grain boundaries are effective barriers to dislocation movement and cause buildup; many slip planes appear to diminish in grain boundary regions. Substructures developed within the grains of polycrystalline alloys affect strength properties and deformation characteristics. Even nominal straining followed by heating to a high temperature can develop these substructures.

The scanning electron microscope (SEM) was utilized as this enabled high-magnification examination of surfaces with good depth of focus. Examination of the 3.8Sn-1.2Ag alloy, Fig. 16(a), shows a very restricted surface movement. Very fine slip bands are evident, as well as the remnant cellular structure from the original solidification of the alloy. An interlocked grain boundary and a very fine slip structure with some evidence of a substructure and very slight folding can be observed in the region of the triple point.

The 3.8Sn-0.5Ag alloy shows considerable variation in the deformation pattern of the surface grains. Figure 16(b) shows multiple slip bands, some kinking, and two prominent folds. Generally the slip pattern is very fine, and many transcrysalline cracks have formed or are in embryo stages, almost normal to a wavy slipped structure within the grains.

Considerable surface deformation was observed in the 6Sn-1.2Ag alloy shown in Fig. 16(c). Numerous needles on the surface are probably contamination of the sample. Multiple slip lines are visible with considerable slide in one direction. The limit of ductility has been reached as shown by the transcrysalline fractures at the ridges. A block slip mechanism appears predominant in this alloy with very little transfer of strain once deformation has commenced.

The surface structure of the 4Sn-2.0Ag alloy is comparatively fine with surface ripples of no particular distinction, as in Fig. 16(d). Surprisingly little surface slip has occurred, yet both transcrysalline and grain boundary fractures are found. The grains have obviously considerable resistance to deformation, with failure occurring on a particularly weak plane. The grain boundary cracking observed does not appear to be associated with any noticeably localized heavy deformation, although some grain reorientation has taken place and slight surface folding is discernible.

Surface examinations after mechanical test have demonstrated that the alloys with relatively low tendencies towards surface slip generally give the highest strength values. However, evidence is presented that lead-tin alloys can be overstiffened by silver additions, which can produce cracking both along and within the grains, resulting in some mechanical strength losses.

All the interface fractures produced very fine dimpled structures.
showing that selected soldering conditions were satisfactory for producing good joints. A typical fracture surface of the bulk solder revealed when the brass is peeled away is presented in Fig. 17(a) for the Pb-3.8 Sn-1.2Ag solder alloy. Figure 17(b) shows the fine, dimpled structure of the Pb-6.0Sn-1.2Ag alloy although the cell size is somewhat larger at this tin content level. However, since the 6.0Sn-1.2Ag alloy actually gave better fracture propagation strength, it appears there is no particular virtue in finer dimpled structures in this general cell-size range.

Metallographic Examination

Compositional changes in the lead-tin-silver system have shown significant effects on mechanical properties of the soldered joint. An understanding of the solder alloy metallurgy must include information gained from metallographic examination by the conventional light microscope. Samples selected for examination were all made at the same soldering temperature of 850°F, rapidly water-cooled after 5 sec exposure with the brass specimen. The direct influence of compositional change can therefore be studied. Figures 18(a) to 18(e) are arranged to illustrate the effect of progressively higher silver additions to a nominally 4% Sn solder, remainder lead.

The base alloy containing 3.8% Sn and no silver is shown in Fig. 18(a), and is seen to be a large-grained single-phase alloy, with some evidence of a developing substructure along the grain boundary. A small addition of 0.5% Ag induces a second phase, as presented in Fig. 18(b). The structure generally consists of large columnar growth normal to the brass surface. A change in morphology should be noted in the region of the interface, where dendrites have rapidly grown parallel to the cooling surface. A substructure of fine grains can be seen developing in this region. A further increase in silver to 0.85% was made as shown in Fig. 18(c). A slight increase in the second phase is noted with some modification of the background matrix. The columnar growth is finer, and the modified structure adjacent to the brass has been reduced in width.

Considerable change in morphology occurs when 1.2% Ag is added, as presented in Fig. 18(d). A third phase has formed, and the solidification pattern does not appear to be quite so directional. The second phase of darker appearance has also become more prominent. Increasing the silver content to 2.0% (shown in Fig. 18(e)) results in a fine columnar structure, a substantial quantity of second-phase, probably a eutectic, and no modified region adjacent to the brass.

In keeping with the philosophy established earlier 1-3 of minimizing the presence of the copper-tin intermetallics, all these microstructures are substantially free from these compounds.

The highest strength lead-tin-silver alloy achieved so far in this program contains 3.0% Sn and 1.5% Ag. The resulting microstructure after exposure at 850°F for 5 sec in contact with brass is shown in Fig. 18(f). A very fine structure of primary dendrites in a eutectic matrix is observed.

To summarize, the microstructures presented have shown a definite relationship between morphology and strength data. Initial appearance of a second phase produces a reduction in strength, which is not recovered until this phase predominates or a third phase is formed. Microstructures obtained with 5% and 6% Sn alloys with approximately 1% Ag show a marked reappearance of the A-phase and consequent reductions in strength values obtained.

Summary and Conclusions

The mechanical properties of conventional and experimental solder alloys have been examined using the tensile-peel test method. Measured strengths of conventional lead-tin alloys differ from those obtained with the lap-shear test and a possible explanation is offered that suggests both a physical and metallurgical basis for the differences.

High lead alloys in the lead-tin system produce the best tensile-peel strengths, and this advantage is retained at elevated temperatures. The addition of silver in a composition 97.5Pb-1.0Sn-1.5Ag results in the best tensile-peel properties of the conventional solder alloys investigated.

Experimental solder alloys in the lead-tin-silver system with compositions of up to 6% Sn and up to 2% Ag were prepared and studied. Higher strength solder compositions in the lead-tin-silver system were found, and the tensile-peel test method proved to be compositionally sensitive. The vital role of solder alloy constitution and morphology in achieving improved strength properties was demonstrated. A good correlation is observed between strength data and the metallurgical structures of the solder alloys. Surface examination of joints after mechanical test has revealed the significance of alloy deformation characteristics on the resulting mechanical properties.

A cost-effective high strength solder alloy has been developed with a composition range of 3.0-3.8 Sn, 1.2-1.5 Ag, and balance lead. This alloy gives approximately 40% fracture initiation strength improvement at room temperature and up to 100% fracture initiation strength increase at elevated temperatures. The new solder alloy also provides some improvement in fracture propagation strength when compared to the 97.5 Pb-1.0Sn-1.5Ag alloy. It is realized that attention must now be given to the determination of the dynamic strength properties of the new solder to determine commercial possibilities for automotive radiator application.

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Fig. 18—Solder morphology after joining to copper alloy 260 (brass). (a) Large-grained single-phase structure of 96.2 Pb-3.8 Sn alloy. (X500, reduced 33%); (b) Two-phase structure of Pb-3.8 Sn-0.5 Ag alloy. (X500, reduced 33%); (c) Two-phase structure of Pb-3.8 Sn-0.85 Ag alloy. (X500, reduced 33%); (d) Dendritic structure with two other phases present in Pb-3.8 Sn-1.2 Ag Alloy. (X500, reduced 33%); (e) Fine columnar structure in Pb-4.0 Sn-2.0 Ag alloy. (X500, reduced 33%); (f) Fine dendritic structure with considerable eutectic present in Pb-3.0 Sn-1.5 Ag alloy. (X500, reduced 33%)

References