

Materials Properties and Associated
Test Methods for Metallic Materials

ESA SME Initiative
COURSE ON MATERIALS AND
PROCESSES

Dr. Ton de Rooij
Head of Materials Mechanics and Processes Section
Materials and Processes Division
Product Assurance and Safety Department

Content of presentation

- ECSS-Q-70
 - Materials properties
 - Constraints on materials
- Surface finishes/coatings
- Joining
- Corrosion testing and Mechanical testing
- Macroscopic and Microscopic examination
- Non-destructive examination
- Failure analysis

ECSS Specification Related to PMP

- ❑ **ECSS-Q-70A-April 96** Space Product Assurance - Materials, Mechanical Parts and Processes.

- ❑ The purpose of this standard is to define the requirements and statements applicable to Materials, Mechanical Parts and Processes to satisfy the mission performance requirements. This standard also provides details concerning the documentation requirements and the procedures relevant to obtaining approval for the use of PMP in the fabrication of Space Systems and associated equipment.

European Co-operation for Space Standardisation (ECSS)

For materials and processes the situation is presently as follows:

- 1. ESA PSS-01-70 has been replaced by ECSS Specification ECCS-Q-70A**
- 2. An additional twenty-four specifications, based on the PSS-01-7xx Series, have been issued as ECSS Specifications**
 - ECSS-Q-70-02A Thermal vacuum outgassing test for the screening of space materials
 - ECSS-Q-70-01A Contamination and cleanliness control
 - ECSS-Q-70-04A Thermal Cycling test for the Screening of space materials and processes
 - ECSS-Q-70-07A Automatic machine wave soldering
 - ECSS-Q-70-08A The manual soldering of high reliability electrical connections
 - ECSS-Q-70-10A Qualification of printed circuit boards
 - ECSS-Q-70-11A Procurement of printed circuit boards
 - ECSS-Q-70-13A The Peel and Pull Off Strength of Adhesives
 - ECSS-Q-70-18A Preparation, assembly and mounting of RF coaxial cables
 - ECSS-Q-70-20A Determination of the susceptibility of silver-plated copper wire and cable to plague corrosion
 - ECSS-Q-70-21A Flammability testing for the screening of space materials
 - ECSS-Q-70-22A The control of limited shelf-life materials
 - ECSS-Q-70-25A The Application of Aeroglaze 2306
 - ECSS-Q-70-26A Crimping of high-reliability electrical connections

European Co-operation for Space Standardisation (ECSS)

- ECSS-Q-70-28A Repair and modification of PCB assemblies
- ECSS-Q-70-29A Offgassing Testing of Materials
- ECSS-Q-70-30A Wire-wrapping of high reliability electrical connections
- ECSS-Q-70-33A The Application of Thermal Coating PSG120FD
- ECSS-Q-70-34A The Application of Aeroglaze H322
- ECSS-Q-70-35A The Application of Aeroglaze L300
- ECSS-Q-70-36A Materials selection for controlling stress corrosion
- ECSS-Q-70-37A Test method for stress corrosion cracking
- ECSS-Q-70-46A General requirements for procurement of threaded fasteners
- ECSS-Q-70-71A Data for selection of space materials
- **An additional seven specifications are in various stages of ECSS review.**
 - ECSS-Q-70B Materials, mechanical parts and processes
 - ECSS-Q-70-05 Detection of organic contamination of surfaces by IR spectroscopy
 - ECSS-Q-70-06 Particle and UV radiation testing of space materials
 - ECSS-Q-70-09 Measurement of thermo-optical properties of thermal control materials
 - ECSS-Q-70-38 High reliability soldering for surface mount and mixed technology PCBs
 - ECSS-Q-70-50 Particulate contamination control in clean rooms by particulate fall-out (PFO) measurements
 - ECSS-Q-70-51 The termination and splicing of optical fibres, fibre optic cables, cable assembly and installation

Metallic Materials used in space

- ❑ Light metals, such as beryllium, magnesium, aluminium and titanium and their alloys
- ❑ Steels, such as low-alloy, tool, corrosion resistant, precipitation hardable, and maraging
- ❑ Nickel and nickel base alloys, including pure nickel, Monel alloys, Inconel alloys, and other nickel- and cobald-base superalloys
- ❑ Refractory metals, principally niobium and molybdenum
- ❑ Copper-base alloys, including pure coppers, beryllium coppers, bronzes and brasses
- ❑ Precious metals
- ❑ Welding, brazing and soldering alloys
- ❑ Various plating alloys

Aluminium and its alloys

Aluminium alloys are some of the basic building materials of existing spacecraft and appear in many subsystems.

Light alloys based on aluminium are used in:

- ❑ primary and secondary structures;
- ❑ plumbing;
- ❑ plating in many applications (electronics, thermal control, corrosion protection etc); aluminised layers on other materials (see 'adhesive tapes' and 'plastic films');
- ❑ fillers in other materials to provide electrical or thermal conductivity.

In addition to standard alloys, more recent alloy developments include:

- ❑ additions of lithium to increase mechanical performance and decrease density. Li-additions are often lower than other 'conventional' alloying elements, so Al-Li alloys may appear within different alloy groups (2000-, 7000- and 8000-series wrought products).
- ❑ reinforced alloys (metal matrix composites - MMC) consisting of aluminium alloys reinforced with whiskers, metal wires, boron fibres or carbon fibres.
- ❑ thin Al-alloy sheets with layers of fibre-reinforced polymer composite in between (Fibre Metal Laminates - FML).

Aluminium and its alloys, cont...

Main categories

A large number of commercial, wrought and cast, alloys are available. A similarly large number of mechanical and thermal tempers are used to optimise certain properties, often at the expense of others (e.g. higher strength, but poorer corrosion resistance). Not all of these alloys or tempers are suitable for aerospace engineering, from the point of view of either mechanical performance or environmental resistance.

Many aluminium alloys exhibit excellent corrosion resistance in all standard tempers. However, the higher-strength alloys, which are of primary interest in aerospace applications, must be approached cautiously. In structural applications preference should be given to alloys, heat treatments and coatings which minimise susceptibility to general corrosion, pitting, intergranular and stress-corrosion cracking. Some alloys are clad with thin layers of pure aluminium to improve corrosion performance.

Copper and its alloys

General

Copper and copper-based alloys are established materials in electrical, electronic and also in more general engineering applications (such as bearing assemblies, etc). Not all are acceptable for space, so discussion is limited to those alloys which have been evaluated and to specific comments relating to their use in space.

Use in spacecraft

The main applications for copper are in electrical/electronic subsystems (wiring, terminals in soldered assemblies) and plating (electronics, thermal control, corrosion protection etc). Copper is also used as a metallising coating -see Plastic Films - and as an additive in other materials - see Lubricants.

Copper and its alloys, cont...

Main categories

Copper materials are generally grouped as:

- ❑ **commercially pure grades**, of which there are many different 'named' varieties that indicate the manufacturing method and the level of control of impurities, including oxygen;
- ❑ **alloys** in which the alloying additions affect the metallurgical microstructure and consequently their characteristics (mechanical, electrical and thermal properties, environmental resistance). The main alloying addition generally provides the named classifications:
 - **brass**: copper - zinc alloys, often containing other alloying elements, such as lead which acts as a 'lubricant' for machining operations - so-called 'free-machining';
 - **bronze**: copper - tin alloys, often containing other alloying elements.

Electronic assemblies use wires made of high-purity copper or copper alloy and terminals of copper alloy.

Beryllium-copper (also known as copper-beryllium) is a copper alloy with small additions of Be. These alloys are used for electrical/electronic applications (spring contacts); for low temperature applications; for high-strength corrosion resistant components and in safety applications in hazardous environments (no sparks produced when impacted).

Copper is also used as a matrix phase in some reinforced metals

Nickel and its alloys

General

As a family, the Ni-based alloys are used in many engineering fields for their corrosion resistance and high-temperature performance.

Ni-alloys are often known by trade names, rather than by their specification code numbers.

- ❑ Some alloys are used in electrical applications (such as heating elements).
- ❑ The magnetic characteristics of certain alloys are utilised in transformer components.
- ❑ A few alloys have controlled-expansion and constant-modulus properties (bimetals, thermostats, glass sealing, precision equipment).
- ❑ Others have been developed for specific applications (hydrogen storage) or to exploit a particular peculiarity (shape-memory effect).
- ❑ There are also a number of alloys used as welding and brazing filler materials. Some Ni-based materials are applied as coatings or hard facings to other materials to provide wear or corrosion resistance.

Nickel and its alloys, cont...

Use in spacecraft

Nickel plating appears in many applications (electronics, thermal control, corrosion protection etc).

Ni-alloys are applied to subsystems requiring corrosion resistance (storage and delivery systems); high-temperature performance, often combined with oxidation resistance (propulsion units - gas turbines and rocket motors, power generation, heat-exchangers and turbines); high-reliability, high-strength fasteners.

Magnetic alloys find a limited but important role. 'Memory alloys' may find use as actuators.

Titanium and its alloys, cont...

Main categories

The characteristics of titanium alloys are generally grouped according to their metallurgical structure which is, in turn, controlled by the chemical composition and heat-treatment history.

- ❑ Commercially pure (CP Ti) products are normally selected for chemical resistance. Impurities in CP Titanium can increase strength but with a loss in corrosion resistance.
- ❑ Titanium alloys are normally selected for their strength properties, which depend on a number of specific heat-treatments (age hardening, quench and temper). The most commonly used titanium alloy is Ti6Al4V for which extensive mechanical and corrosion property data is available.

T i t a n i u m a n d i t s a l l o y s

General

Titanium and Ti-alloys are generally chosen for their mechanical properties, temperature resistance and/or chemical resistance. The specific points of special interest for the spacecraft designer are considered here, since the basic aspects of titanium alloy assemblies are similar to those for aeronautic design.

Use in spacecraft

Conventional Ti-alloys are used for primary and secondary structures; fasteners; in plumbing systems (standard tube alloy grades and commercially pure CP-grades) and in areas where operating temperatures preclude the use of aluminium alloys. 'Memory alloys' based on titanium may find specialised uses as actuators.

Steels

General

Steels, as a family of materials, offer a wide range of characteristics that find uses in many and varied applications. This section concentrates on those materials, normally aircraft grades, which may be considered for use in space and any precautions required for their application.

Use in spacecraft

Steels are used in structural items (e.g. rocket motor casings) and within engineering components (bearings, springs, etc.) in a variety of subsystems and devices

Steels, cont...

Main categories

- ❑ Steels are based on alloys of iron and carbon (between 0.05% and 2%C). All contain some level of other elements, i.e. even plain carbon steels (up to 1.7% C) contain manganese up to about 1%Mn.
- ❑ Impurity levels (e.g. phosphorus and sulphur) depend mainly on the smelting and melting processes used
- ❑ Alloy steels contain one or more additional alloying elements to improve properties and workability.
- ❑ The tensile strength of plain carbon steels increases with carbon content up to approximately 0.8%C, reaching a theoretical maximum of about 900 MPa, with a corresponding decrease in ductility. Hardness increases progressively with C-content, so that low- (0.1-0.3%C) to medium-carbon steels (0.3-0.6%C) are used for various 'engineering' components, whereas high-carbon steels (0.6-0.9%C) are used for applications requiring hardness and wear resistance.

Stainless steels

General

Stainless steels - also known as corrosion-resistant steels - have alloying additions specifically to provide a continuous, adherent, self-healing oxide film and so reduce the attack of corrosive media.

In addition to corrosion resistance, they also exhibit a number of other properties making them useful engineering materials (oxidation resistance, creep resistance, toughness at low temperature, magnetic or thermal characteristics).

This section concentrates on those materials, normally aircraft grades, which may be considered for use in space and discusses precautions required for their application.

Use in spacecraft

Use of stainless steels in spacecraft centre on applications requiring corrosion resistance (e.g. storage and handling of liquids and waste), components within some thermal protection systems and fasteners such as high-reliability, high-strength bolts.

Stainless steels, cont...

Main categories

Stainless steels contain chromium (at least 12%) which provides the protective oxide film, plus a number of other alloying elements to enable a range of characteristics.

- ❑ **austenitic** - derived from the basic 18Cr/8Ni compositions (300-series), or higher strength versions in which some of the Ni-content has been replaced by nitrogen and manganese (200-series). Strength is increased by cold-working and properties are retained at low temperatures.
- ❑ **ferritic** - 400-series materials contain between 11-30%Cr and a maximum of 0.1%C. Often used in the annealed or cold-worked condition, increased strength can be obtained by heat-treatment.
- ❑ **martensitic** - also fall within the 400-series, normally have chromium contents between 11 and 18%. Some can be heat-treated to give high tensile strengths (>1400MPa).
- ❑ **duplex** – mixed ferritic/austenitic microstructures. High Cr and Mo contents provide pitting corrosion resistance and reasonable resistance to SCC in chloride environments, (i.e. better than some austenitic grades).
- ❑ **precipitation hardened** - based on martensitic or duplex grades with additions of copper and aluminium for precipitation hardening. They can be heat-treated to give high strengths combined with high corrosion resistance.

Miscellaneous metallic materials

General

A metal is classed as miscellaneous if it does not fall within another Declared Materials List (DML) category in ECSS-Q70B. Also included in this section are comments on metal-based materials that are either prohibited or should be approached with caution for space applications.

Use in spacecraft

- ❑ Light alloys based on magnesium and beryllium are used in some primary and secondary structures.
- ❑ Plating appears in many applications (electronics, thermal control, corrosion protection etc) and calls mainly for silver and gold.
- ❑ 'Memory alloys' based on titanium and nickel may find uses as actuators
- ❑ In addition to standard conventional alloys, more recent material developments include:
 - reinforced alloys (metal matrix composites - MMC) consisting of magnesium alloys reinforced with carbon fibres;
 - lithium additions to conventional magnesium alloys;
 - reinforced silver alloys.

Miscellaneous metallic materials, cont...

Main categories

Miscellaneous metals include, but are not limited to:

- ❑ magnesium alloys;
- ❑ beryllium and Be-alloys. (See: 'Copper and Cu-alloys' for Be-Cu alloys);
- ❑ refractory alloys;
- ❑ superalloys, which as a group include cobalt-, iron- or nickel-based alloys. (See: 'Nickel and Ni-alloys' for Ni-based superalloys);
- ❑ mercury;
- ❑ plating materials: cadmium, zinc, tin, gold, silver, osmium etc.

This section also includes comments on metal-based materials that are either prohibited or should be approached with caution for space applications.

Filler materials: welding, brazing, soldering

General

Fusion joining techniques produce permanent joints. Soldered joints and some brazed joints can be disassembled with care.

Use in spacecraft

Welding is a common fabrication method for metals used in spacecraft.

Brazing usually refers to joining with alloys of copper, silver and zinc and is preferred to soldering when stronger joints and an increase in temperature resistance is required.

Soldered joints are used for electrical and thermal conducting paths and for low mechanical strength joints. Soldering is commonly referred to as 'soft-soldering' in which low-melting point alloys, such as tin-lead or indium-based materials are used.

Filler materials: welding, brazing, soldering, cont...

Main categories

Filler materials, welding procedures and post-weld processes are detailed in aerospace standards and specifications

Comments on weld filler materials also apply to **braze metals** and processes. An added complication is that braze fillers are generally very different from the parent weld materials and so galvanic couples and other corrosion effects also need consideration.

Solder alloys that are acceptable for use in electronic assemblies in space, and their associated fluxes and process chemicals (solvents; cleaning baths, etc), have been subject to intense evaluation, see the tables '**Guide to choice of solder-types for space use**' and '**Representative products**' table (from ECSS-Q-70-08).

Solder alloys consist of the tin-lead and indium-lead alloys defined in ECSS-Q-70-08 and ECSS-Q-70-38. They are procured according to these specifications, which define purity levels and, where necessary, fluxes of suitable formulation for the assembly of spacecraft electronics.

Materials Properties

- Strength
- Elastic modulus
- Fatigue
- Fracture toughness
- Creep
- Micro-yielding
- Coefficient of thermal expansion and coefficient of moisture expansion
- Stress corrosion
- Corrosion fatigue
- Hydrogen embrittlement
- Wear and fretting
- Surface finishes
 - Anodising
 - Conversion
 - pickling/etching
 - mechanical
- Coatings
 - hard
 - barriers

Constraints on materials

- Temperature
- Vacuum
- Thermal cycling
- Chemical (corrosion)
- Galvanic compatibility
- Atomic oxygen
- Moisture absorption/desorption
- Fluid compatibility

Interfaces (surfaces, layers)

- **Anodising**
 - On Aluminium, Titanium
 - Normal anodising and black anodising.
- **Chemical conversion**
 - As corrosion protection, underlayer for paint
- **metallic coatings (overlay and diffusion)**
 - Al clad layers, Ni, Au, Ag
- **hard coatings**
 - Electroless Ni, Cr
- **Diffusion barriers**
 - Under coatings of Cu, Ni, e.g. between brass and solder
- **Other coatings**
 - High temperature oxidation protective coatings
 - Thermal barriers
 - Moisture barriers
 - coatings on CFRP

Coating, cont..

- Depending on the design and service, the following coatings are used:
 - Paints
 - phosphate coatings
 - chromate coatings
 - electroless nickel
 - chromium, electroplated
 - oxides and black oxide
 - nickel/tin, electroplated
 - tin/lead
 - electroless nickel plus electroplated chromium
 - vapour-deposited aluminium
 - metals, such as gold, silver, palladium, rhodium
 - lubrication coatings such as bray coat grease
 - molybdenum disulfide
 - carbides and nitrides or combination

Joining

- Mechanical fastening
- Bonding
- Combined bonding and fastening
- Fusion, including welding, brazing, soldering and diffusion bonding

Joining cont..

- Fusion techniques can be grouped as:
 - soldering
 - brazing
 - welding, including diffusion bonding
- All fusion methods produce permanent joints. Soldered, and some brazed joints may be disassembled with care.
- All fusion techniques require extensive quality control and inspection procedures.

Joining cont..

- **Soldering**

- Soldering is commonly referred to as soft soldering in which low melting point alloys such as Tin-Lead or Indium based alloys are used. Soldered joints are used for electrical and thermal conducting paths and for low mechanical strength joints.
- Soldering shall not be used for structural applications unless reviewed and approved by the Customer.
- See session by C. Villette

- **Brazing**

- Brazing is preferred to soldering in those cases where a strong joint, which is heat resistant is required. As distinct from soft soldering brazing usually refers to joining with alloys of copper, silver and zinc

- **Welding**

- Numerous welding techniques are available. In the aerospace industry the following techniques are often used:
 - *Tungsten Inert Gas (TIG) welding*
 - *Plasma-Arc welding*
 - *Electron Beam (EB) welding , laser welding*
 - *Resistance welding (induction, spot, seam welding), Diffusion welding*

Corrosion Testing

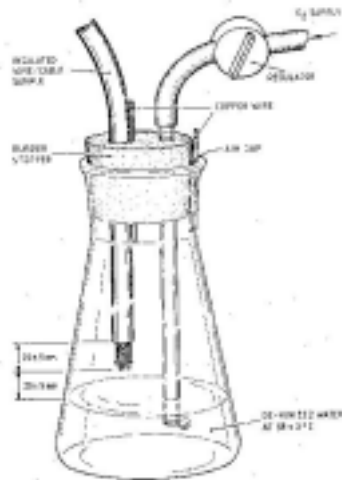
- General Corrosion
 - Passivation
 - Anodising,
 - plating
- Stress Corrosion (see presentation of Dr. G. Bussu)
- Bimetallic Corrosion
 - Good selection
- Atomic Oxygen Corrosion (already presented)
- Red Plague Corrosion
 - The use of ESCC qualified wires and cables

General corrosion testing

Salt-spray-(foreground) and Humidity Chamber- both with temperature and humidity regulators



Red plague testing



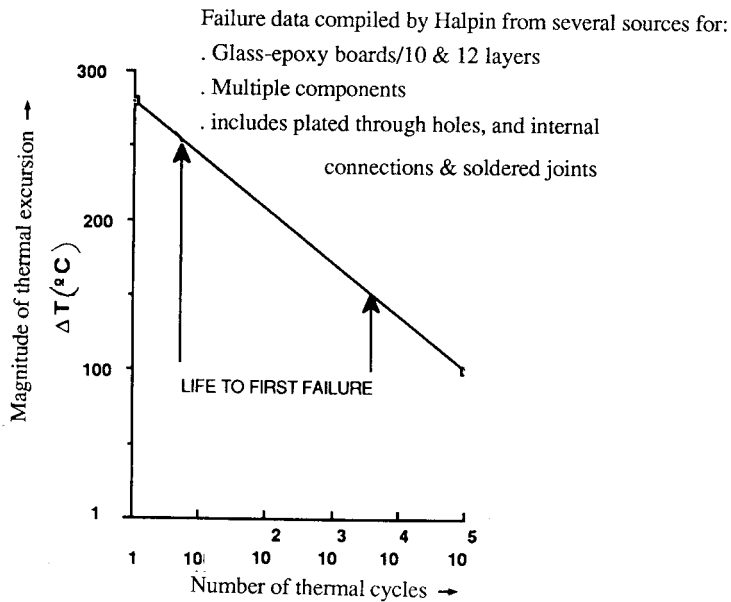
Red plague testing (Anthony and Brown test) is performed according to ESA PSS-01-720

Test conditions: 240 hrs at 58 °C

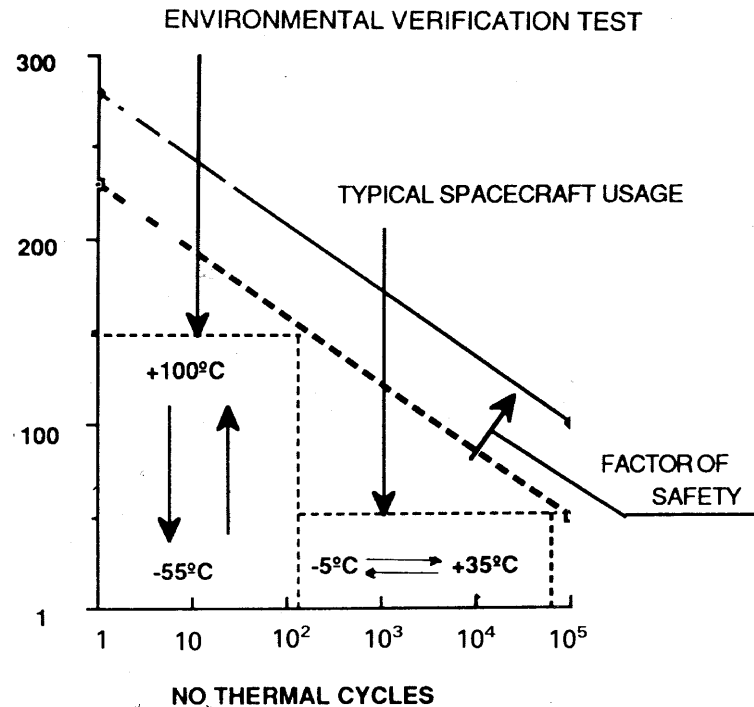
Mechanical Testing

- Hardness testing
- Tensile Testing
- Compression testing
- Fatigue testing
- Fracture Toughness
- Thermal cycling

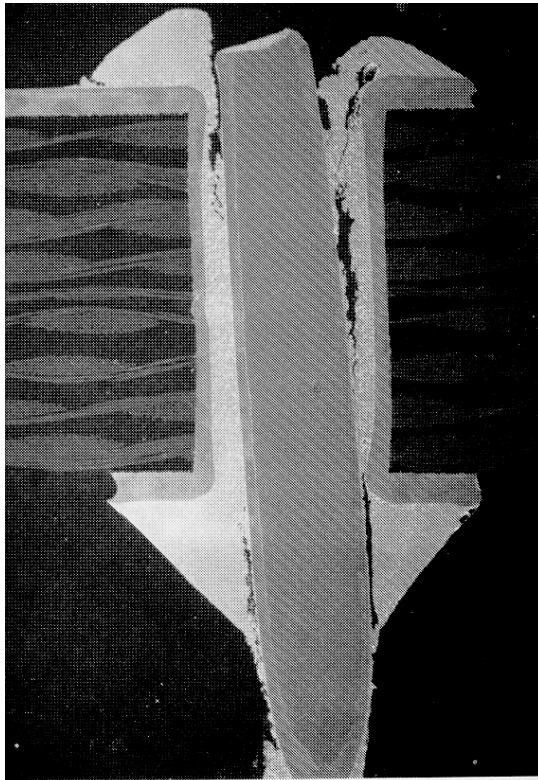
Thermal fatigue



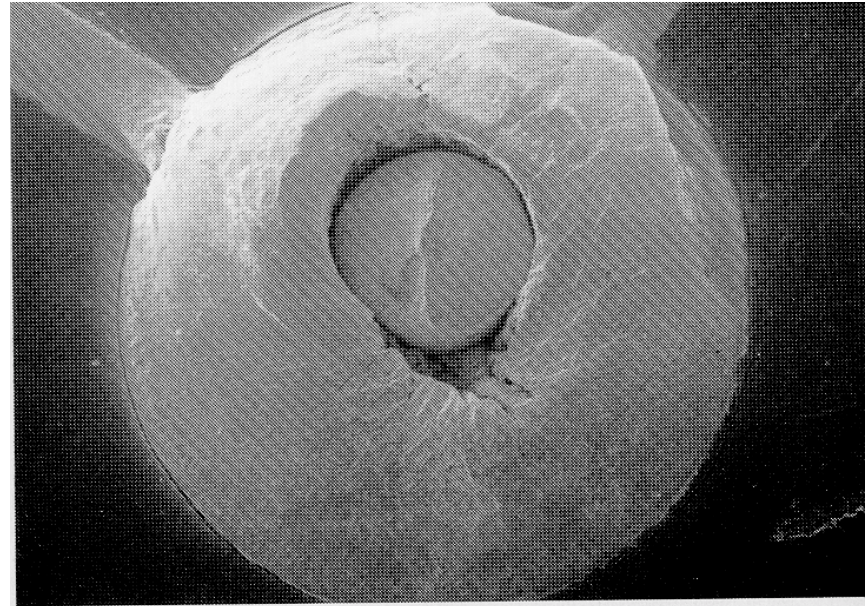
(b) Fatigue curve for printed circuit assemblies from Reference 2



Failures: After thermal cycling



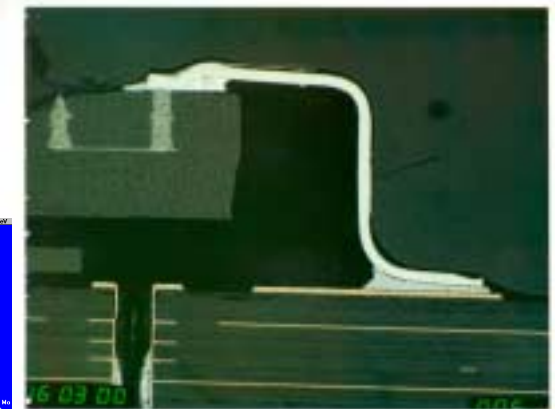
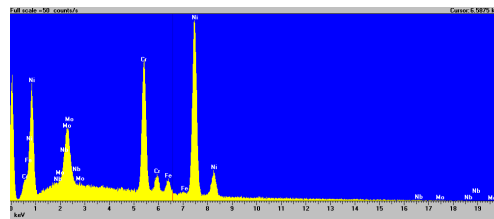
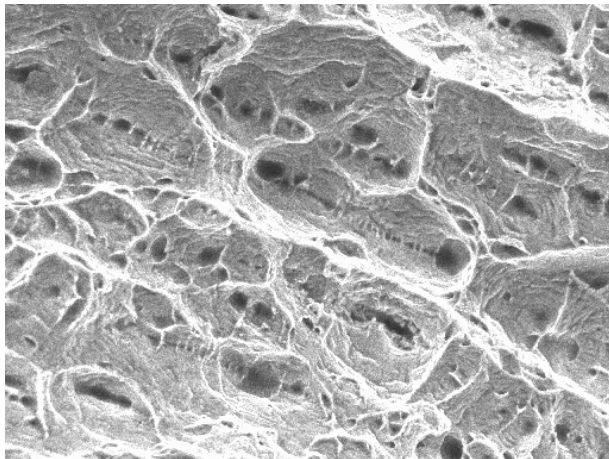
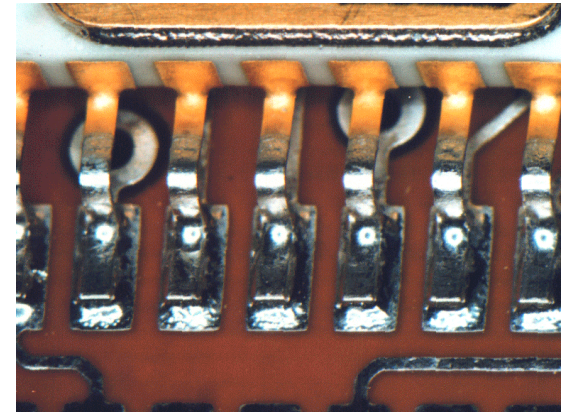
Cross section



Top view

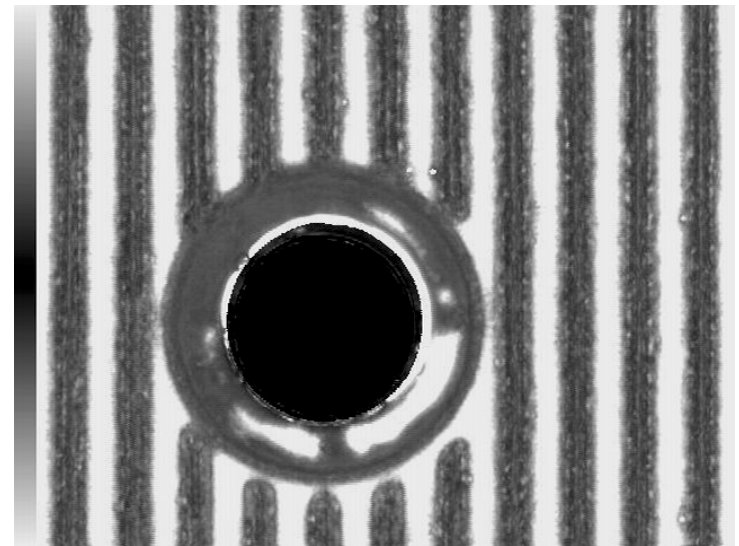
Macro / Microscopic Examination

- Macroscopic (< X75)
- Light microscopy
- Confocal microscopy
- Scanning Electron Microscopy



Non-Destructive Examination

- Visual Inspection
- Dye penetrant
- Eddy Current
- Acoustic Microscopy (SLAM, C-SAM)
- X-Ray



Failure Analysis

Under control of Material Review Board (MRB)

- Collection of background data
- visual examination
- selection, identification, preservation of specimens
- macroscopic examination and analysis
- microscopic examination and analysis
- metallographic sections examination
- failure mechanism
- chemical analysis
- simulated testing
- report