NASA Contractor Report 177932

BASIC CRYOGENICS AND MATERIALS

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New Castle, Delaware

Contract NAS1-17571
June 1985

(NASA-CR-177932) BASIC CRYOGENICS AND MATERIALS. PHASE 1 Supplemental Report (Applied Cryogenics and Materials Consultants) 20 p HC A02/MF A01 CSCL 20L

N85-32215

Unclas
G3/31 21924

National Aeronautics and
Space Administration

Langley Research Center
Hampton, Virginia 23665

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SUMMARY

This paper summarises the effects of cryogenic temperatures on the mechanical and physical properties of materials. Heat capacity and thermal conductivity are considered in the context of conservation of liquid nitrogen, thermal stability of the gas stream and the response time for changes in operating temperature. Particular attention is given to the effects of differential expansion and failure due to thermal fatigue. Factors affecting safety are discussed, including hazards created due to the inadvertent production of liquid oxygen and the physiological effects of exposure to liquid and gaseous nitrogen, such as cold burns and asphyxiation. The preference for using f.c.c metals at low temperatures is explained in terms of their superior toughness and the limitations on the use of ferritic steels is also considered. Non-metallic materials are discussed, mainly in the context of their LOX compatibility and their use the the form of foams and fibres as insulants, seals and fibre-reinforced composites.

1. INTRODUCTION

The industrial production and handling of cryogenic fluids such as liquid nitrogen, oxygen, hydrogen and helium, as well as liquefied natural and petroleum gasses is now based on mature technologies developed and refined over many decades. The needs of a cryogenic wind tunnel using large quantities of liquid nitrogen do not differ significantly from those of, for example, a large chemical plant or food freezing factory and thus much of this technology is directly transferable. It is, however, important to recognise that the majority of those involved in running or using a cryogenic wind tunnel are unlikely to have had previous experience of cryogenic fluids. It is therefore particularly important to ensure that the accumulated experience on the safe handling of cryogenic fluids is also passed on to these new users. Much of this experience has now been gathered together in manuals and texts such as references 1, 2 & 3. This information should be digested and understood not only by those with managerial responsibility for safety, but also by those directly involved, and as far as is practical, by those indirectly involved in the use of cryogenic fluids. Unjustified alarm created in the minds of those in receipt of a suitable training program can usually be allayed by a full and frank examination of the facts. **Justifiable alarm is better exposed before an accident, when remedial action can be taken, than after a tragedy. Finally, the old adage "familiarity breeds contempt" is unfortunately true and even experienced personnel can get careless. Cryogenic fluids such as liquid nitrogen deserve a healthy respect,** but when handled with care, their use can open up new areas of technology such as the cryogenic wind tunnel. In view of the importance of using the correct procedures in the design, construction and operation of cryogenic tunnels, those sections of this paper that have a direct bearing on safety will be highlighted by the use of bold print.

Those involved in the design and construction of cryogenic wind tunnels and the models that are to be tested in them need a more thorough understanding of the properties of cryogenic fluids and materials and techniques of construction. In the previous AGARD lecture series No. 111 on Cryogenic Wind Tunnels, (Ref. 4), the author gave two lectures on the Physical and Mechanical Properties of Materials and Dr. R. G. Scurlock gave three lectures on Cryogenic Engineering. These lectures set out basic principles for the safe handling of cryogenic liquids and the construction of cryogenic equipment and, five years later, these principles are equally valid. In this lecture we will try and distill the essence from the material contained in these five lectures and update it in the light of the progress made since the first lecture course. For a more thorough understanding of the subject the reader is, however, encouraged to consult the original papers, particularly as much numerical data on the physical and mechanical properties of materials was collated in the tables therein (Refs. 5 & 6) Further valuable information is also contained in Toblers excellent report on "Materials for Cryogenic Wind Tunnel Testing" (Ref. 7)

Before considering these factors in detail, it is worth taking a brief overview of a typical large installation. Firstly, let us consider the storage and transfer of the large quantities of liquid nitrogen needed to run a tunnel. In principle, this is virtually identical to the situation which exists in, for example, a large food freezing plant. The storage vessels, pumps, valves and control equipment, all serve the same purposes and there are, therefore, sound reasons for considering them as a commercial package once the relevant design specification has been established. Thus, for example, it should not matter whether 9% nickel steel, 304 stainless or 5083 aluminium is chosen for the construction of the LIN storage vessel as long as it is carried out by a technically competent organisation. In many respects the design and construction of the transfer line should also be a relatively simple commercial consideration once local constraints and requirements have been identified.

Secondly, in the design and construction of the tunnel itself it is necessary to bear in mind the extra constraints that cryogenic operation will introduce. For example:

- thin, light structures cool down more rapidly and evaporate less cryogenic fluid than do heavy sections, thus, if fast thermal response is required it is essential to minimise the thermal mass of the structure.

- insulation is necessary to cut down the heat inleak to the working space and hence the effective refrigeration power used. This insulation can be applied either internally or externally and the implications of this decision are manifested in considerations of the smooth profile of the inner liner in the first case and in the toughness of the pressure shell at cryogenic temperatures in the second.

- all materials contract to a greater or lesser extent when they are cooled and one of the essential aspects of the successful design of cryogenic equipment lies in avoiding the problems created by differential contraction caused by temperature gradients or the juxtaposition of dissimilar materials.

- some materials embrittle at low temperatures and it is of critical importance to select materials with strengths and toughnesses adequate for their intended duty. The failure of even a non-structural component could possibly cause damage further down the tunnel, or lead to the premature end of a test run.

- all materials used must be compatible with their working environment both internally and externally. Design must ensure the prevention of accidental condensation of liquid oxygen, particularly in the presence of hydrocarbon based polymers which are LOX incompatible.

Thirdly, it is important that designers and operators are aware of the differences that a low temperature environment will induce in a tunnel and its associated equipment as compared to conventional operation at ambient temperatures. Thus, certain aspects of the model suspension and force measuring systems will have to be reconsidered in the light of their cryogenic operating environment, for example:

- the materials used to construct the sting assembly have to be very strong and stiff. In many alloys high strengths are associated with low toughnesses and as the strengths of all metals increase at low temperatures, it is essential to ensure that their toughness does not fall to unacceptably low levels: current state of the art technology seems to favour the various grades of maraging steel and the precipitation hardened and high-nitrogen forms of stainless steel for sting construction.

- if the force balance systems are to operate at ambient temperature in a cryogenic tunnel, heaters must be used to warm the appropriate regions. Low conductivity materials have to be used to provide the necessary heat breaks between warm and cold regions, while high conductivity inserts can even out unwanted temperature gradients.

- alternatively, if the whole system is to operate at low temperature it has to be possible to calibrate out the variations in the gauge constants brought about by changes in the electrical resistivity of the metallic films or wires and adequate moisture proofing is essential.

- provision should be made for the removal of the model assembly from the test section without the need to warm up the whole tunnel. Furthermore, a cold model assembly should be allowed to warm up in an atmosphere of dry nitrogen if problems caused by moisture condensation and frost build up are to be avoided.

At this stage it is worth emphasising that care needs to be exercised in the use of data taken from compilations and reference manuals because some properties are more "structure sensitive" than others. For example, the electrical and thermal conductivities, strength, ductility and toughness of materials are properties that are highly dependent on the microstructural and chemical condition of the material. In contrast, the specific heat, thermal expansion and elastic moduli are relatively unaffected by the presence of structural defects. Thus, although it is possible to apply the data taken from the literature for the structure-insensitive group of properties, it would be unwise, and even dangerous, to use uncritically the values given for the defect sensitive properties. These should be used for guidance only and if at all possible, they should be backed up by data obtained experimentally on material obtained from the suppliers of the batch of material to be used: in the absence of such experimental verification, generous safety margins should be applied to the literature data.

2. THERMAL AND OTHER PHYSICAL PROPERTIES OF MATERIALS

2.1 Heat Capacity and Specific Heat

Information on the heat capacity or specific heat of materials used in the construction of cryogenic equipment is necessary in order to calculate the energy that has to be supplied for cool-down to the operating temperature. Structures with the highest heat capacities require the largest amount of cooling and this has to be supplied by the latent heat of the evaporating liquid or by the sensible heat of the cold gas. For structures which have to undergo frequent cooling and warming cycles, it is important to minimise the total heat capacity, or thermal mass, to achieve both low liquid boil-off rates during cool-down and also short cooling times: for equipment that rarely warms up once it is cooled, low heat capacities are not so important. A further, and highly relevant, example of the effect of thermal mass may be illustrated by comparing the operating experience of the NASA 0.3-m TCT with that of the tunnel at the University of Tsukuba, Japan. In the NASA tunnel virtually no problems were experienced in controlling the temperature of the working gas by varying the liquid nitrogen injection rate, while the Japanese group found the maintenance of steady temperatures much more difficult. The clue to this difference is to be found in the designs of the tunnel liner and insulation system. The NASA tunnel is insulated on the outside of the 6061-T6 aluminium alloy pressure shell and thus a large thermal mass of metal is cooled down to the working temperature. The thermal inertia of this large mass evens out fluctuations in the gas temperature that would otherwise be created by variations in the liquid nitrogen injection rate. In the Tsukuba tunnel the insulation is inside the mild steel pressure shell and the inner wall is thin and has a low thermal mass. It is thus unable to absorb much heat without its temperature rising and the liquid nitrogen injection control system has to work much harder to achieve temperature stability. On the other hand, deliberate changes in the operating temperature are achieved more rapidly in the Japanese tunnel.

For heat balance calculations it is, in fact, the enthalpy, $H = \int Cp\ dT$, which is of most direct use and in Reference 8 tabulated values of the enthalpy relative to absolute zero are given together with the specific heat at constant pressure, Cp, for a range of metals and non-metals. The specific heats of all materials drop off at low temperatures eventually to become zero at 0 K, and the very low values found at hydrogen and helium temperatures can cause large temperature differences to be set up by a small heat-influx. At liquid nitrogen temperatures and above these effects are not so severe.

Although large amounts of cold work may cause a slight decrease in heat capacity, for practical purposes specific heats are largely unaffected by the normal range of conditions found in metals. The specific heats of pure crystalline solids over the complete temperate range is given by the Debye theory and knowledge of the characteristic temperature, θD, allows calculation of the specific heat at the required temperature (Ref. 9). Specific heats of alloys at room temperature are given approximately by the Kopp-Newman rule of mixtures in which the specific heat of a metallic solution is given by the sum of the products of specific heat and molar fraction for each constituent element. Although the rule gets less applicable at low temperatures, in the absence of alternative data it gives an acceptable first approximation. Furthermore, it is worth noting that the lattice structure has a strong influence on specific heats as illustrated by the observation that the measured specific heat of f.c.c. austenitic stainless steels are closer to those calculated for gamma iron than those measured on the b.c.c. alpha iron. The specific heats of non-crystalline and amorphous materials cannot be described by the Debye theory and there is, therefore, no satisfactory alternative to measured values for materials such as glass and amorphous ceramics, as well as all polymers, elastomers, composites and adhesives. When considered on a unit mass basis most of these materials have high heat capacities compared to metals, but this discrepancy is reduced if they are considered on a unit volume basis.

2.2 Thermal Conductivity

Conduction of heat in solids takes place through the vibration of their lattice atoms, and in the case of metals, by the movement of their conduction electrons. Any mechanism which makes these processes more difficult lowers the thermal conductivity of the material and hence high conductivities are found in pure, strain free, large grain or single crystal metals and non-metals, while low conductivities are associated with impure, stressed, amorphous or microcrystalline structures. As it is difficult, if not impossible, to recognise these different conditions by looking at the surface of a material, and as the physical and mechanical history of the sample is rarely well documented, great uncertainties can arise in using thermal conductivity data from the literature. However, in many cases, conductivities at one extreme or the other are required - for example, very low conductivities where heat breaks are required to reduce heat influx, or very high conductivities to minimise thermal gradients. In general, good conductors are materials of high purity and in an annealed state, while bad conductors are either alloys with many components and complex microstructures, or non-metals with amorphous or microcrystalline structures. Still lower conductivities may be obtained by increasing the number of interfaces crossed by the heat flux. For example, stacks of stainless steel discs may be used for compressively loaded, thermally-insulating supports, while the combination of many fine glass filaments with a thermo-setting plastic matrix (G.R.P) gives a material with the highest known ratio of tensile or compressive strength to thermal conductivity. The use of G.R.P. supports to separate the inner and outer skins of modern vessels for storing cryogenic liquids is, in a large measure, responsible for the low boil-off rates currently achieved.

It should, however, be noted that although the amorphous or microcrystalline structures of most non-metals make them very efficient thermal insulators, it also makes them very brittle, especially in the bulk form and they can be excessively prone to thermal shock if cooled rapidly. Furthermore, variations in their density, structure and processing history can change their thermal conductivities by about an order of magnitude as well as causing considerable anisotropy, so care has to be taken in extracting suitable values from the literature.

2.3 Thermal Expansion

This is probably the most important of the physical properties because the stresses set up in components by differential thermal expansion can very easily cause severe distortion or, at the worst, failure. The total linear contraction of a number of representative materials is shown as a function of temperature in Fig. 1. It can be seen from the figure that the total linear contraction at 77 K varies from about 0.05% for Invar and Pyrex glass to over 2% for some thermosetting resins, and it is not surprising, therefore, that problems can arise when materials are used together without adequate forethought. Problems can, in practice, usually be resolved into two basic categories:

i) those in which only one type of material is involved and where differential contraction is a result of temperature gradients,

ii) those in which the same temperature gradient is applied across two or more materials of different expansion coefficient.

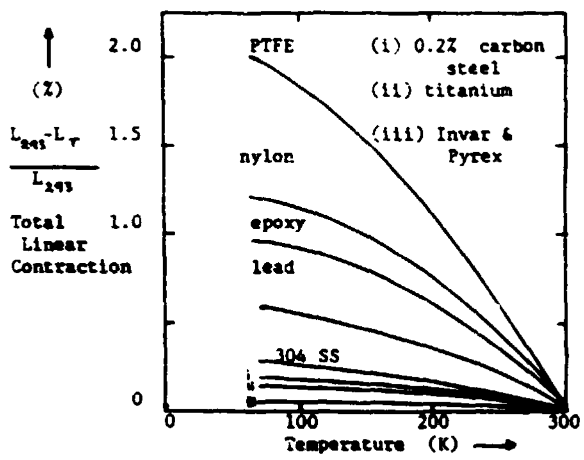


Figure 1. Total Linear Contraction of Selected Materials

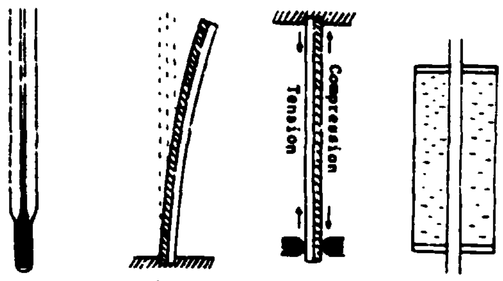
Considering first the case of dissimilar materials, a common mercury in glass thermometer uses the large differences in expansion coefficients between the two components, but no stresses are set up as the mercury is free to move inside the glass tube (Fig. 2a). In contrast, a bi-metallic strip consists of two metals firmly fixed together, and when the temperature decreases the free end moves towards the side containing the metal with the higher expansion coefficient (Fig. 2b). If the end were not free to move the metal with the higher expansion coefficient would be put into tension and the other metal into compression (Fig. 2c). An idea of the forces that can be set up by contraction in dissimilar metals can be obtained by considering the hypothetical arrangement illustrated in Fig. 2(d), in which co-axial copper and steel pipes joined at both ends are cooled to 80 K. The total linear contraction of copper is 302 $\times 10^{-5}$, while that of a 0.2% carbon steel is 192 $\times 10^{-5}$, a difference of 110 $\times 10^{-5}$ or just over 0.1%. Thus the differential strain is slightly larger than that considered to give the 0.1% proof stress, which in copper at 80 K is about 88 MPa. If the joint between the two metals were a soft lead-tin solder it would have to yield and flow in order to accommodate this degree of mismatch. (Data from Ref. 10)

An even more relevant example is illustrated in Fig. 2(e) which shows a section of an externally-insulated, closed-circuit cryogenic tunnel. When cold the wall of the tunnel contracts relative to its warm mountings and, as one end is effectively clamped by the fan shaft bearing, the other end must be able to move to prevent thermally-induced stressing on cooldown. In the NASA LaRC 0.3-m TCT this is accomplished by supporting the wall on a stainless steel supports which slide on re-inforced PTFE pads. Differential contraction between the inner and outer walls is a common design problem in transfer lines for cryogenic fluids and some form of expansion joint has to be built into the system. It was noted earlier that the total linear contraction of Invar from 300 to 77 K was very much smaller than other alloys, about 1/6th of that of austenitic stainless steels and 1/8th of that of aluminium alloys. Thus a transfer line with the inner wall made from Invar would need only 1/6th or 1/8th as many expansion joints as it would if made from stainless steel or aluminium alloy respectively and the savings thus achieved are sometimes more than enough to offset the higher material and fabrication costs associated with Invar.

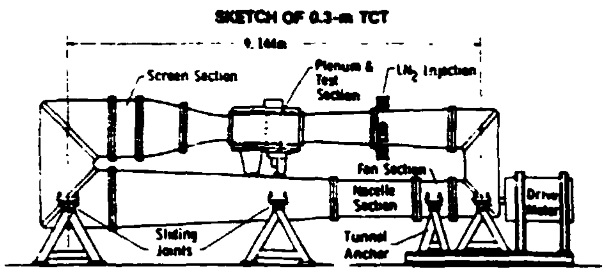
A further example of mismatched materials is illustrated in Fig. 2(f) by a flanged joint between aluminium alloy and stainless steel pipes. Aluminium alloys contract more than stainless steels and if an aluminium alloy bolt were used its loading would be increased as it contracted more rapidly than the stainless steel flange. It is possible that the bolt might in fact fail on cooling: if not it would yield and stretch so that on warming to room temperature it would now be too long to compress the gasket adequately and a room temperature leak would be created. The use of a stainless steel bolt would also cause problems because on cooling it would contract less rapidly than the aluminium flange and so be unable to keep the same compressive stress on the gasket - the likely outcome being a low temperature leak which would then seal itself up when the joint were rewarmed to ambient temperature. This type of low temperature leak will be recognised by those with cryogenic experience as a source of considerable frustration!

One elegant solution to this problem is shown in Fig. 2(g). A long stainless steel bolt passes through the centre of a Monel compensating sleeve as well as through the two flanges, the length of the Monel sleeve being calculated to compensate exactly for the lower contraction in the bolt. The total linear contractions at 80 K relative to 293K are 391 $\times 10^{-5}$ for aluminium, 236 for Monel and 285 for type 304 stainless respectively, hence the difference between the stainless bolt and the aluminium flange is 106 $\times 10^{-5}$ and that between stainless and Monel is 49 $\times 10^{-5}$. If the aluminium flange were 10 mm thick a Monel sleeve 10 x106/49, i.e. 21.6 mm long would be needed for exact compensation. The same principle may be used for joints between 9% nickel steel and aluminium flanges by using an Invar (Nilo 36) sleeve to compensate for the contraction in the 9% Ni steel bolt.

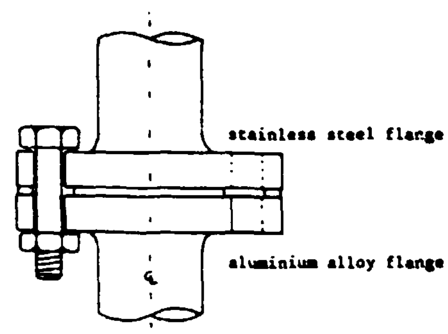
Returning to the case where temperature differences can cause problems even when the material is the same, Fig. 2(h) shows schematically a situation in which co-axial, thermally-insulated vessels are joined at their extremities. If the vessels were made of mild steel the total linear contraction of the inner shell at 80 K would have been 192 $\times 10^{-5}$ relative to the outer shell which remained at ambient temperature.



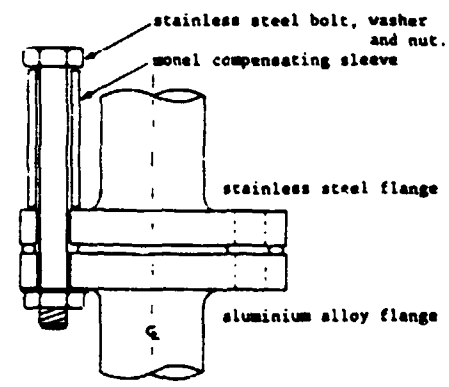
2(a) 2(b) 2(c) 2(d)



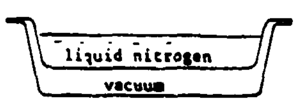
2(e)



2(f)



2(g)



2(h)

Figures 2(a) to (h) Examples of Differential Thermal Contraction caused by Dissimilar Materials or Temperature Gradients.

This strain was too large to be accommodated by the mild steel which was not only below its ductile-brittle transition, but in all probability embrittled by the welding used in its fabrication.

The whole question of fits and clearances at low temperatures has to be kept very much in mind. Most of us are familiar with the practice of heating a gear wheel before placing it onto a shaft so that it will shrink to a tight fit on cooling. Some will also be aware that the same operation is sometimes carried out by cooling the shaft with liquid nitrogen prior to fitting the gear so that the required fit is obtained when the shaft expands on rewarming to room temperature. These examples should be remembered when constructing models, balances or other fittings where there are close fits and small clearances. On cooling these clearances could either decrease and cause a seizure, or increase and lead to looseness and possible leakage. This can also manifest itself in changes in the clamping force applied to models which could decrease on cooling and allow the model to vibrate loose, or increase and possibly cause damage. Reference to Fig. 1. will remind us that these problems are likely to be particularly severe where non-metallic materials are involved as their total linear contractions are so large.

Finally it is worth reiterating the comment made earlier about thermal shock. We have now seen that most non-metals have low thermal conductivities and high expansion coefficients, and we will find later that many of them also become embrittled at low temperatures. We thus have a combination of the three factors that lead to thermal shock and they are particularly severe if the materials are present in thick sections and/or cool-down rates are high. Nevertheless, brittle materials can be used safely at low temperatures if enough care is taken. For example liquid hydrogen bubble chambers have plate glass windows for viewing ports which are cooled at a rate of a few degrees K per day to prevent thermal shock. In the case of viewing ports for cryogenic wind tunnels, it is probably much better to follow the practice adopted in the prototype NASA tunnel of adopting quadruple glazing purged with dry nitrogen. This not only minimizes thermal shock but it cuts down the heat loss and prevents condensation on the outer skin. When purging a multi-layer system it is important to ensure that the purge gas is fed in from the warm si   and exhausted at the cold face, as flow in the opposite direction is liable to cause condensation on the outer layers as they are cooled by the cold gas being fed from the inside.

3. PROPERTIES OF CRYOGENIC FLUIDS

The production of tonnage quantities of liquid oxygen and nitrogen by the fractional distillation of liquid air is a commercial process that has been developed continuously over almost 100 years. The availability of liquid nitrogen in tonnage quantities initially came as a bye-product of the requirement for large quantities of liquid oxygen for use in steel making, rocket fuels and other applications. Liquid nitrogen is readily available and relatively inexpensive and it was this combination that triggered the initial development of the prototype cryogenic wind tunnels in the early 1970's. Modern large tunnels such as the NTF consume so much nitrogen that a dedicated air separation plant is needed for their supply, the liquid oxygen now being the saleable bye-product. Although the designer or operator of a cryogenic wind tunnel does not need to know the details of the commercial liquefaction process, some understanding of the basic thermodynamic mechanism of the separation of liquid air into its major constituents is desirable as incorrect design or operation of equipment that uses liquid nitrogen can cause the inadvertent production of liquid oxygen and create a potentially serious fire hazard. Basic aspects of Cryogenic Engineering are described in references 11 and 13.

3.1 Liquid Air, Oxygen and Nitrogen

The basic properties of liquid air and its constituents are set out in Table 1 and discussed in the next two sections.

Table 1 Properties of Liquid Nitrogen, Air, Argon and Oxygen (Refs. 1 and 11)

Property	Nitrogen	Air	Argon	Oxygen
Molecular Weight	28	28.8	40	32
Critical Pressure (atm.)	33.5	38.7	48.3	50.1
Critical Temperature (K)	126	132	151	154
Normal Boiling Point (K)	77.4	Bubble 78.8, Dew 81.8	87.3	90.2
Freezing Point (K)	63.2	-	84	54.8
Liquid Density at Normal Boiling Point (kg/m3)	808	876	1402	1138
Specific Gravity of Gas at 288 K and 1 atm.	0.97	1	1.38	1.10
Vol. Gas @ 288K & 1 atm. /unit vol. liquid @ B.P.	683	730	823	843
Latent Heat of Vaporisation (kJ/kg)	199	205	161	213
Specific Heat of Liquid Cp, (J/kg.K)	2.038	1.967	1.138	1.699
Liquid Viscosity (microPascal.sec)	158	163	256	188
Paramagnetism	none	oxygen / 5	none	strong
Colour	colourless	light blue	colourless	blue
Oxidizing Power	none	moderate	none	very strong

3.1.1 Binary phase diagram for oxygen-nitrogen mixtures

The binary phase diagram between pure oxygen, B.P. 90.2 K, and pure nitrogen, B.P. 77.3 K, is shown in Fig. 3. The composition of gaseous air is taken as 21% oxygen, 79% nitrogen, the minor constituents such as argon being ignored for the sake of simplicity. For air the dew point temperature, where droplets of liquid start to condense from the saturated vapour, is 81.8 K. The bubble point temperature, where bubbles of gas start to form in the saturated liquid, is 78.8 K. The horizontal tie-line drawn at 81.8 K connects the composition of the vapour, 21% oxygen-79% nitrogen, with that of the liquid with which it is in equilibrium, 50% oxygen-50% nitrogen, thus illustrating that the liquid is enriched with oxygen. In commercial air separation this enrichment is exploited by re-evaporating the liquefied air and allowing the nitrogen-enriched gas to rise up the column while the liquid descends and becomes progressively richer in oxygen as more and more nitrogen evaporates.

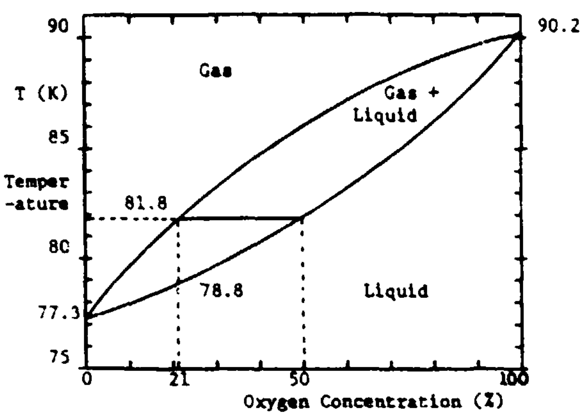


Figure 3. Nitrogen / Oxygen Phase Diagram

3.1.2 Inadvertent Liquid OXygen (LOX) formation.

It is, however, the inadvertent formation of oxygen-enriched liquid that is of much greater significance to the operator of a cryogenic wind tunnel. If air comes in contact with a surface cooled to temperatures below 81.8 K, it will condense and form a liquid enriched in oxygen. Subsequent evaporation of the nitrogen will further enrich the liquid until the remaining liquid is virtually pure oxygen. This can constitute an extremely serious fire hazard if there are combustible materials present. All hydrocarbon-based solids, liquids and gases are LOX incompatible and the greatest care should be taken to avoid their presence in an oxygen-enriched atmosphere. If the cold surface is visible and covered with frost, its temperature is too high to condense liquid oxygen. If it looks wet and free of frost it is probably because the condensing liquid air has washed any frost away.

Despite its low temperature, liquid oxygen is an extremely efficient oxidizing agent and many materials, including some metals, will burn violently if ignited in its presence as the heat released during combustion is about an order of magnitude greater than the latent heat needed to vaporise the liquid to gas. Particularly reactive metals such as titanium and magnesium are a hazard even in the bulk form, while ferritic and austenitic steels, aluminium and zinc will burn fiercely when in the finely divided form of dust or fibres. All hydrocarbons, including ordinary clothing, human hair and tissue as well as many of the plastic foams and fibres used in insulation systems are LOX incompatible materials. Further details of the LOX compatibility of plastics materials are given in Table 10 of Ref. 5. There are two ways in which such materials may be used safely. The first is to apply an impervious vapour barrier to the outside of the insulation to prevent air ingress. This has the additional benefit of excluding water vapour which might otherwise lead to ice formation and degradation of the insulation material. The second is to ensure that the material is continuously purged with a dry, inert gas such as nitrogen. In practice these two techniques are best combined by gas purging the space inside the vapour barrier. A further point to note is that, although the appropriate measures may have originally been taken to prevent LOX condensation, subsequent servicing or modification may result in the incomplete re-establishment of an effective vapour barrier. In other cases, especially where the operatives have changed, potential hazards have arisen when LOX-incompatible materials have been substituted for the original, correctly-specified material. A further potential hazard can arise where control valves are hydraulically actuated if the inevitable fluid leakage from old installations is allowed to contaminate the insulation system or, as often happens, is allowed to saturate the flooring material. The combination of these saturated materials and oxygen-enriched air drifting down from an improperly insulated nitrogen-cooled surface would be a serious combustion hazard should they be inadvertently ignited. (Ref. 12).

3.2 Physiological Effects of Nitrogen and Other Safety Considerations

3.2.1 Cold Burns

Despite the apparent contradiction in terminology, the physiological effect of the exposure of human flesh to cryogenic temperatures is similar to that of a thermal burn. The affected tissue dies. In a controlled form this effect is utilized in cryosurgery to destroy unwanted growths and cancers. Even more unpleasant effects are caused if moist, bare flesh is held in contact with a very cold surface, for example an uninsulated pipe carrying liquid nitrogen. The moisture on the skin is frozen hard to the surface and it may be impossible to release the skin without tearing or cutting off the frozen layer. Non-absorbent clothing should be worn when handling cryogenic liquids and care taken to ensure that any spilled liquid cannot be trapped inside shoes. Gloves should be dry, non-absorbent and loose-fitting so that they could be removed rapidly if liquid got inside. Should a cold burn occur, flowing cold water should be used to thaw the affected area.

3.3.2 Oxygen Deficiency, Anoxia or Asphyxiation

It is only necessary for the oxygen content of breathing air to fall a few percent below its normal value of about 20% for bodily functions, both mental and physical, to be adversely affected, hence the use of oxygen breathing sets for climbing mountains and high altitude flight. Reduction of the oxygen level towards about 14% causes anoxaemia which is characterised by an increase in pulse rate, laboured breathing and difficulty in concentration. At oxygen levels between 14 and 10% the victim is still conscious but muscular effort causes rapid fatigue and mental processes such as co-ordination and judgement deteriorate. When the oxyg : concentration falls below 10% there is a severe risk of asphyxiation and possibly permanent brain damage. By the time the victim realises that something is wrong it may be too late for him to save himself as his muscles will be unable to function and allow his escape. If the oxygen level falls below 6% death is virtually inevitable - apparently painless, but nonetheless permanent! It is, in fact, surprisingly easy to achieve such low oxygen concentrations. Inhaling just a few breaths, or even one deep breath of pure nitrogen, or any other inert gas, can flush the oxygen out of the lungs and the loss of muscle function can prevent them refilling even if the victim is removed from the inert atmosphere. Some form of rapid resuscitation would be necessary to restore oxygen to the lungs and allow possible recovery. A typical scenario for such an accident is where someone opens and inspection hatch in a nitrogen-purged vessel, puts his head inside to "take a quick look" for something only to collapse within a few seconds because his lungs have become filled with nitrogen. Little or no warning is given by the body of this form of anoxia, unlike the gradual loss of breathable air that takes place in a sealed volume when the oxygen is not replaced.

There are two important areas in which the effects of anoxia can be avoided. Firstly, it is necessary to be able to detect the presence and extent of regions of low oxygen concentration. Oxygen monitors have replaced the traditional canary for this purpose and used correctly they are invaluable. Care is, however, needed in their location. If, for example, they are placed too high up they will not register a dangerous loss of oxygen at working head height. Placed directly over a nitrogen vent or on the floor below an outlet they will trigger prematurely. Such false alarms are likely to lead to distrust or complacency that could prevent operatives from reacting to a truly dangerous situation. Pits and ducts are particularly hazardous as cold gasses tend to sink and accumulate at low levels. When using liquid nitrogen it is essential to maintain a flow of fresh air, often simply by opening the appropriate doors and windows, to prevent the build up of an inert gas.

The second area involves the provision of the appropriate equipment for dealing with an emergency. Particularly important is an advance evaluation of the likely mode and extent of a possible spillage and the measures that should be taken to minimize its effect. For example, evacuation routes should be marked and kept clear. Breathing equipment should be kept handy and personnel properly trained in its use so that they could reach safety and/or effect rescue even in the event of a large spillage and severe nitrogen build-up. Alternatively, the availability of a breathing set could allow someone to remain safely in the affected area to permit rapid remedial action that could prevent a small incident from becoming a major accident. Thus, although automatic shut-down of pumps and closure of valves should be designed into a liquid handling system wherever possible, the ability to close back-up valves manually could also be an advantage in some situations.

. STORAGE AND TRANSFER OF LIQUID NITROGEN

4.1 Heat-Transfer into Cryogenic Liquids

Energy has to be expended in liquefying cryogenic fluids such as nitrogen and heat influxes need to be reduced as far as possible in order to minimise the rate at which it re-evaporates. It is important to realise that even after a cryogenic fluid has absorbed enough heat to overcome the latent heat of vaporisation, additional thermal energy is needed to warm up the gas. Furthermore, the amount of "sensible heat", as it is called, required to warm evaporated nitrogen gas to room temperature is approximately equivalent to the latent heat. In good cryogenic design practice this sensible heat is used to cool radiation shields, entry pipes and other sources of heat-inleaks and thus reduce the net heat flux that is absorbed by the latent heat.

4.1.1 Insulation Systems

A schematic liquid nitrogen storage vessel is shown in Fig. 4. and there are three mechanisms by which heat reaches the cryogenic fluid: conduction, radiation and convection. Consider first conduction. This comes mainly from from heat flowing along the load-bearing supports and connecting pipes and it is minimised by using thin sections of materials with low thermal conductivities such as glass reinforced plastics and cold-worked stainless steels, etc. Many non-metallic materials used for thermal insulation at low temperatures are in the form of finely divided powders, fibres, films or foams and their low conductivities arise not only from the inherent low conductivity of the material, but even more so, from the poor thermal contact between adjacent particles or layers. Further improvements can be achieved in powdered or fibric insulation systems by removing the gas from between the layers and so cutting down convection losses. In the case of insulating foams, it is important to appreciate the role played by the gas or vapour trapped in the cells. If the blowing gas has high melting or boiling points it may be possible to solidify this gas at low temperatures, reduce convection within the cells and thus improve its insulation value. However, if the cells are not completely closed, gas or vapour may permeate from the warm to the cold faces. Not only will this lower the efficiency of the insulation but permeation of water vapour will break down the cell structure by cyclic freeze-thaw action. As noted earlier, an even more serious problem can be caused by the permeation of air through imperfect foam insulation surrounding liquid nitrogen cooled surfaces as this can lead to preferential condensation of liquid oxygen and the creation of a potential combustion hazard. The solution to both of these problems is to provide a efficient vapour barrier on the warm side of the foam to prevent the ingress of gas or vapour, and this also helps to minimise ageing problems. Closed cell foams are widely used for the thermal insulation of liquid nitrogen and other cryogenic systems. They are relatively cheap, efficient and easy to apply, some being foamed in situ. Other types of foam, particularly the extruded type of polystyrene slabstock, have good load bearing characteristics - in general the strongest foams have the highest densities and the highest conductivities.

Radiation, particularly the infra-red component, is reduced by the use of heat-shields, which are either actively cooled by contact with the evaporated, cold gas or act passively by increasing the number of radiating layers between ambient and and cryogenic temperatures. Absorption of radiation at the liquid surface is sometimes reduced by a layer of floating spheres called Kroffels which reduce the area of liquid that "sees" higher temperature radiation. In the most effective systems of all, the super-insulants, thin metallic films intercept the infra-red radiation and chemical getters are use to soak up any residual gas.

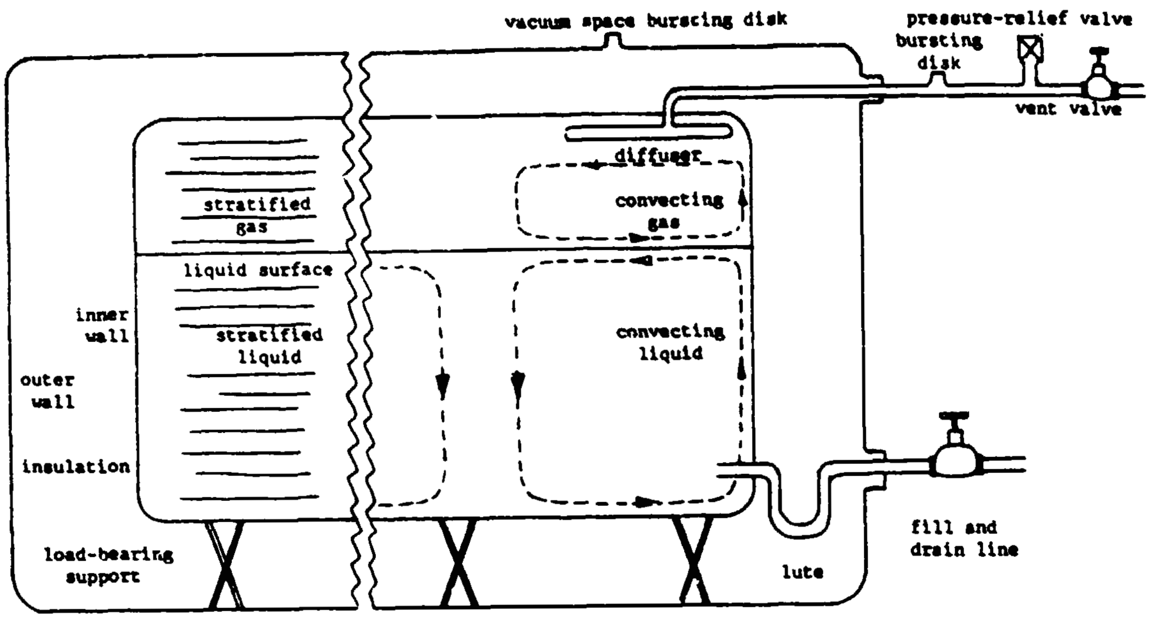


Figure 4 Schematic Representation of Aspects of Liquid Nitrogen Storage

4.2 Storage of Liquid Nitrogen

Storage vessels for cryogenic liquids such as nitrogen are not designed to be full of liquid and about 10% of the total volume, called the ullage space, is left above the liquid surface. This space also allows liquid to separate out and settle as the vessel is filled and in a typical transfer, once the vessel is about 90% full, liquid appears in the gas stream venting from the vessel indicating transfer is effectively complete. Even with an efficient insulation system, there is always a net influx of heat reaching the cryogenic fluid in its storage vessel and this evaporates gas which collects in the ullage space. If the heat influx is minimal, stable stratified layers build up in both liquid and gas with density gradients that tend to oppose any natural convection, as inferred on the left hand side of Fig. 4. For larger heat influxes the gas and liquid in contact with the wall are warmed causing them to rise and set up separate Convection systems in both the liquid and the gas. Consider first the gas. The walls are hotter than the gas and so absorb some of its sensible heat, thus becoming cooled. Correct design should enable all of the incoming heat flux reaching the unwetted wall to be absorbed by the cold gas so that there is no heat flow down to the wetted walls where it would have to be absorbed by the latent heat of the liquid. The warming gas rises by convection in the boundary layer adjacent to the walls and falls again in the centre to flow outwards across the liquid surface sweeping with it the cold evaporating gas, so completing the convection cycle, as indicated in the right hand side of Figure 4.

4.2.1 Storage Instabilities

Similar convection cycles exist in the liquid, with upward flow in the boundary layer at the walls and then across the surface where it absorbs heat from the counter-flow of gas and evaporates from the surface of the superheated liquid. This surface evaporation, or boil-off as it is often called, has an irregular nature and takes place in cells whose locations move over the surface. If, however, the liquid is left undisturbed for long periods, the boil-off rate decreases and a stable layer of highly superheated liquid develops on the surface. When subsequently disturbed, this superheated layer evaporates suddenly, leading to a rapid increase in the boil-off rate and a rise in pressure. In smaller-sized storage vessels the temperature of this superheated layer can sometimes rise by up to 40 K before explosive boil-off takes place. Condensation of liquid oxygen or argon in the surface layer can take place if air can leak into the vessel and, as these liquids are both hotter and denser than liquid nitrogen, rapid boil-off occurs if the surface layer is disturbed. It is, in fact, considered good practice to stir liquid nitrogen slowly to prevent the build-up of unstable stratified layers.

4.2.2 Provision of Relief Valves and Bursting Disks.

In Table 1 it was noted that if 1 litre of liquid nitrogen was evaporated and warmed to room temperature and 1 atmosphere pressure it would create approximately 680 litres of gas. If such evaporation were to take place in a enclosed volume the resultant pressure would thus increase to over 600 atmospheres should the structure be strong enough to withstand such a pressure. In practice no storage vessels, transfer lines or other components would be stressed to this level and they would therefore rupture, possibly explosively. To prevent such a hazard, enclosures that could possibly become over-pressurised have to be fitted with relief valves and bursting disks set to trigger and vent the gas safely. It should be noted that the capacity of these items needs to be adequate to cope with the maximum gas flow rate and that the bursting disk should be fitted between the enclosure and the relief valve. Vacuum spaces between the inner liquid container and the external shell must be fitted with bursting disks as a guard against sudden failure of the inner container and consequent ingress of liquid into the insulation. One spectacular rupture of a cryogenic pressure vessel not fitted with a relief valve occurred in Apollo 13 on its way to the moon when an electric heater was accidentally left on in the titanium alloy liquid helium storage vessel. Other more common causes include the blockage of liquid hydrogen and helium storage vessels by the condensation and solidification of air and the plugging of liquid nitrogen vents and lines by condensed and frozen water vapour. The lute shown in Fig. 4 fitted in the fill and drain line between the inner vessel and the valve helps to prevent localised evaporation of liquid from the warm pipe and the possibility of blockage due to the build-up of impurities such as dissolved water vapour and carbon dioxide.

4.3 Transfer of Liquid Nitrogen

4.3.1 Removal of Liquid from the Storage Vessel

There are three principle methods used to remove liquid from the storage vessel (1) self pressurization of the inner vessel, (2) external gas pressurization and (3) pump transfer.

The liquid flow rates obtainable with self pressurization are relatively low and this technique is most frequently used for smaller sized vessels and laboratory scale applications. Some of the liquid is removed from the storage vessel and passed through an external vapourising coil where it evapourates, expands and causes the pressure to increase. This warm gas is fed back into the storage vessel through the diffuser which, together with stratification in the gas prevents the warm gas flowing directly into contact with the liquid and recondensing.

External pressurization can utilise either the same gas as that liquefied in the storage vessel, or a separate, often non-condensable, gas. Considerably higher transfer rates can be achieved by external pressurization together with rapid response times. In the Douglas Aircraft Company 4ft blowdown cryogenic tunnel, liquid nitrogen from a 9000 gallon capacity run tank was pressurised to 300 psia using compressed air stored in separate tanks. Transfer rates of 1440 litres/second (300 gallons/second) of liquid were achieved giving a maximum run time of the order of 30 seconds.

Both the 0.3-m TCT and NTF at NASA Langley used pumps to transfer their liquid nitrogen, 3 pumps being run in parallel when the maximum flow rates of about 450 kg/sec (5.5×10^{8} litres/sec) are needed by the NTF. It should be noted that a Net Positive Suction Head (NPSH) must be maintained at the pump inlet if cavitation is to be avoided. Furthermore as the evaporation rate during liquid transfer is to low to maintain the pressure in the ullage space above atmospheric, nitrogen gas has to be returned to the ullage space to prevent possible collapse of the inner vessel.

4.3.2 Transfer Lines

As noted earlier structures with low thermal mass cool more rapidly and evaporate less cryogenic liquid than those with larger thermal masses and this is one factor that has to be taken into consideration in deciding whether to insulate transfer lines or to leave them bare. When liquid oxygen or pressurised liquid nitrogen is passed through an uninsulated pipe, frost builds up on the outside and tends to insulate it by preventing convective cooling by the air. If, however the nitrogen is subcooled, condensation of liquid air tends to wash away the frost and heat losses are higher. Nevertheless short lengths of bare pipe are often used to transfer liquid nitrogen from delivery tankers to small storage vessels as they are cheaper and less cumbersome than insulated lines. For longer runs and permanent installations transfer lines are, however invariably insulated. An evacuated double walled construction, with or without powder or superinsulation, is usually favoured for the larger and longer lines and, as noted earlier some form of expansion joint has to be provided to prevent the build up of tensile stresses on the inner pipe during cool-down. Flexible foams, with suitable vapour barriers to prevent LOX condensation are frequently used for shorter, smaller runs.

4.3.3 Liquid Transfer

In a perfectly insulated, pre-cooled pipe cryogenic fluid would be transferred as a single phase liquid, but in most practical cases some degree of two phase flow is usually present. This can take many forms depending on whether the pipe is horizontal or vertical, the mass flow rate, pressure drop across the line and heat inleaks to the line. In general, stratified flow with the liquid at the bottom of a horizontal pipe and vapour above it occurs at low flow rates, but at higher flow rates, shear between gas and liquid sets up waves or plugs which can completely fill the pipe with liquid. Under some circumstances annular flow occurs and this can be beneficial if it can be arranged in such away that the gas is on the outside completely surrounding a central core of liquid, as in this case heat inleaks from the pipe walls will be absorbed by the sensible heat of the gas not the latent heat of the liquid.

During initial cooldown the first liquid introduced into a warm pipe evaporates on contact with the warm sides. This gas then flows ahead of the advancing liquid front precooling the walls as it progresses towards the outlet, a process that can often take a surprisingly long time. Increasing the liquid delivery pressure can be an expensive way of speeding up the process as less of the sensible heat of the gas is used in precooling the pipe. Furthermore, as the evaporated gas occupies a much greater volume than the liquid, gas velocities can be very high and frequently the flow is choked at the exit during practically the entire cooldown and leaves at sonic velocity. If, however, the flow resistance in the pipe is large it may be impossible to make the liquid front advance more than partially along the pipe such that liquid never emerges from the other end and a zero delivery condition is obtained. The usual solution to such a problem is to modify the line by installing a sufficient number of intermediate venting points to allow the liquid front to be advanced progressively by venting from these intermediate points until liquid emerges and then moving on sequentially to the next vent point. Unpredictable two phase flow through the liquid nitrogen injectors of a cryogenic wind tunnel can also create problems with its temperature control, particularly if the condition does not occur evenly with all injection positions. One design that overcomes this problem is to feed all injectors from a circular "ring-main" of liquid as this prevents vapour building at any particular location.

Control of nitrogen flow through the injectors is achieved using servo-controlled valves, digital valves being used in the 0.3-m TCT but the more conventional gate and globe valves being preferred for the NTF. Rapid and accurate control of the liquid flow is obtained when the system is cold, but care should be taken during cool-down to prevent rapid opening or closing of valves that allow liquid to enter warm areas. The sudden build-up of pressure from the vaporising liquid could lead to pressure surges and even flow reversal, with the risk of damage to pumps and other equipment in the line. Even emergency vent or stop valves should be arranged so as to operate over a few seconds rather than slamming shut.

5. STRENGTH AND TOUGHNESS OF METALS AT LOW TEMPERATURES

One of the principal design requirements of any piece of equipment is that it should have adequate stiffness, strength and toughness to withstand safely any load or stress that may be applied to it. Operation at low temperatures effectively increases the requirement for adequate toughness at the operating temperature, as virtually all materials are both stiffer and stronger at low temperatures than at ambient. A load-bearing structure must, therefore, be able to cope with not only the static and dynamic stresses which can be predicted for normal operation, but also the thermal shocks it may be subjected to on cool down, the thermal stresses induced by differential expansion during warming and cooling cycles, as well as the accidental overstresses or impact loads that it may receive in the presence of the scratches and dents it is liable to suffer during service.

Most materials are designed to operate within their elastic limits and typical stress and deflection formulae require the use of appropriate values for the elastic constants such as the Youngs, Shear and Bulk modulii and Poissons ratio. Fortunately, the elastic constants are relatively insensitive to structure variations such as changes in grain size, the degree of cold working, heat treatment and small compositional variations etc., while decreasing the temperature in general increases Youngs modulus by about 10% between 300 K and 80 K. Accuracies greater than about 1% are rarely required in the calculations normally used to avoid buckling failure (elastic instability) or excessive elastic deformation (jamming) and thus values taken from the literature can be used with a reasonably high degree of confidence. The design problems created at ordinary temperatures by the relatively low modulii of aluminium alloys when compared to either austenitic or ferritic steels are also encountered at low temperatures where stiffness is important, for example, where shell bending is a significant design limitation, as in a column subjected to a lateral air flow loading. In such cases, the higher modulii offered by, for example, the austenitic stainless or 9% nickel steels would allow either stiffer structures for the same section or thinner sections for the same stiffness.

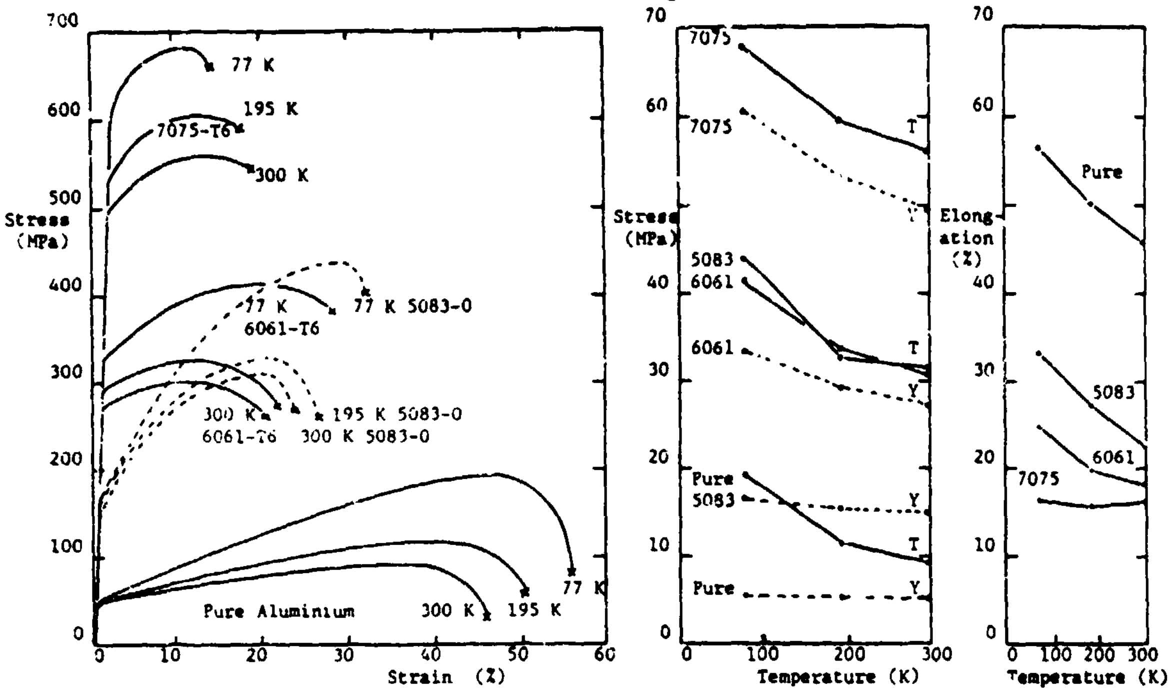
Yield and plastic deformation in metals are processes strongly influenced by their structure and condition to such an extent that accurate predictions are difficult and experimentally determined data invaluable. General classification of their properties is best started by considering their crystal structures, most metals and alloys having face-centred-cubic, body-centred-cubic or hexagonal-close-packed lattices. Of these the face-centred-cubic metals are greatly to be preferred for low temperature use as almost without exception their strengths, ductility and toughness all improve as the temperature falls, thus making them ideal for cryogenic applications. F.c.c. copper-, nickel-, and aluminium-based alloys, together with Invar and the austenitic stainless steels are, in fact, the metals most widely used for the construction of equipment operating below about 150 K. Of the hexagonal-close-packed metals only magnesium and titanium are used in significant quantities below room temperature, the high specific strengths offered by titanium alloys being particularly attractive for certain specialised applications in the aerospace industry.

It is the body-centred-cubic group of metals that offer the greatest challenge but which constitute the greatest risk as they almost all undergo a transition from ductile to brittle behaviour at some temperature, usually below ambient. Furthermore, small changes in their chemical composition, grain size, the degree of plastic constraint brought about by a notch or flaw, and even the rate at which a load is applied, can all have a marked effect on the delicate interrelationship between strength, toughness and the temperature at which the ductile-to-brittle transformation occurs. As, however, the economically irreplaceable ferritic steels have b.c.c. structures, their use at low temperatures cannot be precluded and it is necessary to define the temperature and stress limits to which a certain grade, thickness and condition of steel may be safely used. These limitations are traditionally laid down by codes of practice issued by independent bodies such as the American Society of Mechanical Engineers, government agencies such as the British Standards Institution, local or national insurance agencies or even state regulatory authorities, and there are very few load bearing structures which can be built without conforming to one or more such codes. There is also, however, an increasing and very welcome tendency towards backing up these codes by a scientifically rigorous failure analysis based on the concepts of fracture mechanics - a study of the resistance offered by a material to the continued propagation of a crack nucleating in the vicinity of a sharp crack. Such analyses are also applicable to high strength alloys with f.c.c. and h.c.p. structures, as well as to the rate at which cracks propagate during fatigue and so constitute a powerful analytical technique which the aerospace industry in general was quick to develop and exploit.

The mechanical properties of materials at low temperatures are considered in detail in References 5, 7, 14 and 15, while data is presented in References 16 to 19.

5.1 Face-Centred-Cubic Metals and Alloys

Although some copper and nickel based alloys are used for particular applications in cryogenic wind tunnels, the quantities involved are small and they are best considered in the next lecture in the context of their use in model construction. It is only aluminium alloys and the austenitic iron-based alloys that are likely to be used in significant quantities.



(a) Stress-Strain Curves (b) Stresses vs Temperature (c) Elongation

Figures 5(a), (b) and (c). The Mechanical Properties of Aluminium and its Alloys at Low Temperatures.

5.1.1 Aluminium Alloys

It is instructive to start by considering the properties of pure aluminium because it shows very clearly the basic characteristics of f.c.c. metals which make them so useful at low temperatures. In Fig 5(a) a series of engineering stress-strain curves are shown for aluminium and some of its allo-- ' :ed at and below 300 K. The following features should be noted from the curves for pure aluminium

i) Yield is a gradual process and the yield stress is only a weak function of tempera

ii) The strain hardening rate (slope of the stress-strain curve after yield), the ultimate tensile stress (maximum point in the curve) and the total plastic elongation all increase as the temperature falls. Thus the metal becomes both stronger and more ductile at low temperatures essentially because it is able to accommodate a greater degree of strain hardening before the onset of necking (plastic instability).

iii) The large drop off in measured stress between the onset of necking and final failure is indicative of a large reduction in area and hence a very ductile type of fracture at all temperatures.

In Figs 5(b) and (c) the data is cross-plotted to show more clearly the temperature dependence of the yield and tensile strengths and the elongation. It is the combination of increase in ductility and tensile strength, together with the relative temperature-insensitivity of the yield stress that is the definitive characteristic of face-centred-cubic metals and alloys which makes them so eminently suitable for use at low temperatures. It is brought about by their ability to slip and deform to prevent the build-up of high stress concentrations at the tips of cracks and flaws.

Pure aluminium has high electrical and thermal conductivities and it has replaced copper in many instances where these characteristics are required. Its alloys are widely used where moderately high strengths combined with high toughness and low density are necessary, such as in road and rail transporters for liquid gases, as well as in static liquid storage tanks. Aluminium alloys may conveniently be divided into two groups according to the basic metallurgical strengthening mechanism involved: (1) the solution-hardened alloys, which are very ductile but only moderately strong in the annealed state (although their strengths can be improved by cold working), and (2) the precipitation-hardenable types, which can be heat treated to give considerably higher str..gths.

Of the solution-hardened types, those containing manganese as the main alloying addition (3000 series) have only moderate strengths, but they are very ductile and hence easily formed. Type 3003 is used for tubes, bends, junctions, plate fin heat exchangers, tube plates and trays, as well as in distillation columns and many other applications. Their role in heat exchanges arises largely because they can be dip-brazed in molten salt baths using aluminium-silicon eutectic alloys. They can also be extruded and cut with roller cutters, characteristics which are advantageous for volume production. They can be cold-worked for higher strengths, but, as they are so often used in the welded or brazed condition where these advantages would be lost, it is more usual to use one of the higher-strength alloys where necessary.

The aluminium-magnesium alloys (5000 series) have higher strengths than the 3000 series and they are widely used for the construction of land-based storage tanks, road and rail transporters and for the primary containment of liquid natural gas in LNG ships. A series of stre:s-strain curves from tests at

300, 195 and 77 K on a 5083 alloy is also shown in Fig 5(a) It can be seen that the yield strengths are higher than those of pure aluminium but still relatively independent of temperature. The strain hardening rate is also higher and becomes still more so at lower temperatures so that both tensile strength and elongation improve at low temperatures and thus the alloy is ideal for low temperature use. Type 5083 alloy is, in fact, the largest-tonnage alloy in cryogenic service largely due to its combination of moderately high strength with excellent weldability. Gas-metal-arc and tungsten-metal-arc systems have been used to weld plates up to 175 mm thick; even in the as-welded condition its full strength is retained thus giving it an advantage over the higher strength 2000 and 7000 heat-treatable alloys if post-weld heat treatment is not possible. Additional strength can be achieved in the 5000 series alloys by cold-rolling but the consequent loss of ductility is not always acceptable. Most of the internal structure of the NTF is fabricated in 5000 series alloys. The contraction, test section, high speed diffuser and upstream nacelle are made from welded 5083, as in the non-cryogenic operating mode the temperatures of these parts does not exceed 66 C (150 F). This is the maximum temperature at which 5083 can be operated continuously in a highly stressed state while avoiding the possibility of grain-boundary stress corrosion. Due to the increase in temperature of the gas stream created by the power of the fan, which can be as much as 14 C (25 F) at the high power ambient temperature operating condition, those sections between it and the cooling coil, the downstream nacelle, shrouds and rapid diffuser, are made of type 5454. This alloy can be used safely at the higher temperatures out, as its yield stress is about 50% of that of 5083, thicker sections are necessary.

Aluminium-magnesium-silicon alloys (6000 series) have the lowest strengths of the heat-treatable types and, in general, they are the only ones used outside the aerospace industry. Type 6061 in the T6 condition is stronger than the 3003 and 5083 alloys but its strength in the as-welded condition drops below that of the solution hardened alloys. It is generally available in the forms of pipe, pipe fittings, extruded tubing and other shapes. Stress-strain curves for 6061-T6 are also shown in Fig 5 (a) and it can be seen that their shapes differ from those of pure aluminium and the solution hardened 5083. The yield stresses of 6061-T6 are higher and more temperature dependent and the strain hardening rates are lower. The tensile strengths do not greatly exceed the yield strengths and elongations are smaller at all temperatures than for 5083. Nevertheless, the mechanical properties of 6061-T6 make it suitable for cryogenic applications and it was used for construction of the pressure shell and internals of the NASA LaRC 0.3-m TCT.

The aluminium-copper alloys (2000 series) have higher strengths than the Al-Mg-Si types but their toughness, especially their notch toughness, begins to fall seriously at low temperature and they are not widely used. Moreover, they are often considered to be less easy to weld reliably. Nevertheless, welded type 2014-T6 was employed in the construction of the liquid-oxygen and liquid-nitrogen fuel tanks for the Saturn V Rocket where its high strength/weight ratio proved advantageous.

The final series of stress strain curves in Figure 5 (a) is for one of the very high strength aluminium-zinc-magnesium heat-treatable alloys, 7075-T6. As may be seen, very high yield strengths are attainable at and below room temperature. However, it is also apparent that the ductility, which is already rather low at room temperature, falls even further at low temperatures. If the samples were notched and/or loading rates higher, the picture would appear even blacker as these very high strength alloys have low notch-toughness and are thus rarely used below room temperature. They do, however, provide a convenient example of how a very high strength alloy, even with an f.c.c. structure, can have such a low fracture toughness as to make it necessary to use fracture mechanics analyses for safe design. Even though the alloy does not fail by cleavage, slip is so inhibited by the metallurgical process used to create its high strength that it is no longer able to deform easily and reduce stress concentrations at the tips of cracks and flaws.

5.1.2 Austenitic Stainless Steels

This is one of the most important class of materials used in the construction of equipment for operation at low temperatures. The face-centred-cubic gamma phase of iron is normally only stable at high temperatures, but the addition of alloying elements such as nickel, manganese, carbon and nitrogen suppresses the gamma-alpha transformation and enables the austenitic gamma phase to be retained to room temperature and below. The gamma structure is, however, only metastable and under certain conditions of stressing and/or cooling a partial transformation to martensite can take place in some of the less highly alloyed steels such as type 304. Two martensite phases are formed having h.c.p. and b.c.t. structures respectively, and it is the b.c.t. form which leads to an increase in strength but loss of toughness in the transformed state. Furthermore, b.c.t. martensite is ferromagnetic and its presence is a severe disadvantage in applications where magnetic fields are present. Finally, to make matters even worse, the transformation is accompanied by a volume expansion which can spoil the fit of accurately machined components such as shafts and flanges.

In general, the most stable steels are those with the highest nickel contents such as the 25-Cr, 20-Ni type 310 wrought alloy and the analogous casting alloys CK20 and Kromarc-55. One disadvantage of 310 is its rather low yield strength of about 200 MPa at room temperature, but if some loss of ductility can be tolerated this may, however, be raised to about 500 MPa by cold working without either a significant drop in toughness or transformation to martensite. Because of its high nickel content type 310 is more expensive than other 300 series stainless steels and it is somewhat more difficult to obtain in a wide range of product forms than the more popular alloys. In most other 300 series austenitic stainless steels some degree of martensitic transformation may be induced by stressing or thermal cycling. The ferromagnetic nature of b.c.t. martensite allows its existence to be established very simply by the use of a small pocket magnet, while a somewhat more sophisticated, and hence expensive, variation on this theme uses a calibrated spring balance to measure the force required to remove a magnet from the surface of the sample.

In a series of experiments carried out by the International Nickel Company, the magnetic permeability of a series of austenitic stainless steels was measured before and after cold rolling at 300K and 77 K. It was found that type 310 showed no transformation while type 301 was strongly affected by both treatments. The samples of 316, 321 and 347 showed an increasing tendency to transform during cold rolling but were relatively unaffected by thermal cycling. Type 304, one of the most widely available of all the austenitic stainless steels, has the greatest variability in its behaviour because small decreases in the carbon and nitrogen contents can be very harmful as both elements are strong austenite stabilisers. It is therefore worth noting that the recently developed 'Hi-proof' grades of 304, 316 and 347 which contain 0.2% nitrogen to increase their yield strengths by about 70 MPa, will also be very much more resistant to martensite transformation than the normal grades. In contrast the low carbon grades of 304 used to prevent weld decay will be least resistant to martensitic transformation! When the nitrogen bearing grades 304N, 316N and 347N become more readily available in the product forms required, they will offer probably the best combination of properties available for low temperature applications from any austenitic stainless steel and their use should be encouraged. Regrettably, they are, at present, difficult to obtain in small quantities and in forms other than sheet and plate.

A further point worth noting from the results of the International Nickel Company experiments concerns the effect of thermal cycling. If the ferromagnetic nature of a partially martensitic 300 series steel is not a drawback for a particular application which demands good dimensional stability, it is possible to ensure that transformation is complete before final machining. This may be achieved by cycling repeatedly between 300 K and 77 K, if necessary taking measurements after each few cycles, until no further significant dimensional changes occur. Further consideration of dimensional instability, particularly in the context of wind tunnel models, will be given in the next lecture.

Type 304 is the most readily available of the austenitic stainless steels and the low carbon welding grade 304L was used for the outer pressure shell of the NTF. Under normal operating conditions the outer shell is unlikely to be cooled more than a few tens of degrees below room temperature, due possibly to localised degradation of the insulation. Furthermore, should some malfunction lead to the build up of liquid nitrogen inside the shell, the inherent toughness of the austenitic structure ought to ensure that no embrittlement problems would be encountered.

5.2 Body-Centred-Cubic Metals and Alloys

As a general rule metals with b.c.c. crystal structures undergo a ductile-brittle transition which occurs at some temperature which may be above or below room temperature. There are a few exceptions, but for practical purposes the potentially brittle nature of b.c.c. metals at low temperatures puts a severe limitation on their use. However, they cannot be disregarded as they include the whole range of carbon, low-alloy and nickel steels which are economically irreplaceable for the construction of equipment operating at moderately low temperatures. The basic philosophy behind their successful application lies in ensuring that they have adequate toughness at their minimum operating temperature for them to withstand not only their design stresses but also accidental impact and other overloads without failing in a brittle and catastrophic manner.

5.2.1 The Ductile-Brittle Transformation in Ferritic Steels

One manifestation of the ductile-brittle transformation in low-alloy ferritic steels can be observed in the sharp decrease in the tensile elongation that occurs at the transition temperature. A much more realistic indication of the seriousness of the problem is, however, obtained from the tough-brittle transformation measured by the decrease in the impact energy absorbed in Charpy V notch tests carried out over the transition temperature range. For a plain carbon steel the toughness transition takes place at or above room temperature, whereas the ductility transition occurs at some 220 C lower. About half of this drop can be accounted for by the very much higher strain rates involved in an impact test, as ferritic steels are highly strain rate sensitive. Thus in practice ferritic steels should not be subjected to sharp blows or impact loading when they are below their toughness transition. The remainder of the decrease is due to the presence of the stress concentration at the notch root, an indication of the importance of avoiding sharp corners and minimising stress concentrations. Small variations in the depth or sharpness of the notch also account for much of the scatter in the impact energies measured over the transition range. In general most metallurgical and other factors which strengthen the material, e.g. cold work, increased alloy additions, precipitation hardening, etc., also lower its toughness. The only exception to this rule is the action of grain refinement as this increases both the strength and the toughness. Indeed grain refinement is one of the most important methods of obtaining toughness in ferritic steels at low temperatures, and this is achieved in part by increasing the manganese concentration relative to that of carbon, high Mn/C ratios giving finer grained structures. The degree to which the steel is deoxidised, or killed, by the addition of silicon and aluminium is also important, fully-killed steels being tougher, but more expensive, than semi-killed steels. Niobium and vanadium are also added to high grade steels to produce additional grain refinement, while careful control of the aluminium, nitrogen and vanadium concentrations can lead not only to enhanced grain refinement but also to precipitation hardening by their resultant nitrides and carbides.

It is, however, the nickel alloy steels that are most widely used at and below 220K. In Britain 2.25, 3.5, and 9% Ni steels are readily available and these grades adequately span most required temperature ranges. 2.25% Ni steel is used down to 210 K, particularly for equipment handling liquid propane at 230 K, while 3.5% Ni steel down to 170 K and is commonly specified for tanks, pipes, and other applications involving liquid ethylene, ethane, acetylene and carbon dioxide. It is, in fact, also often employed for higher-temperature applications because the additional safety margins given by its higher toughness compensate for its marginally higher cost compared to 2.25% Ni steel. A 5% Ni steel is also used quite widely in Europe at temperatures down to 150 K, particularly in the fabrication of welded vessels for the handling and storage of liquid ethylene. The use of 9% nickel steel in thicknesses up to 50 mm and at temperatures down to 77 K without post-weld heat-treatment has been allowed since 1962 under ASME code case 1308. This steel is available in both (1) the quenched and tempered, and (2) the double normalised and tempered conditions, (1) having a marginally higher yield stress and lower ductility at room temperature: it is unusual in that post-weld heat treatment actually lowers its toughness and this treatment is therefore not recommended. It is the only ferritic steel permitted for use at liquid-nitrogen temperatures and it is economically competitive for the construction of large storage tanks for liquid nitrogen, oxygen, argon and methane. Its high proof-stress/tensile-stress ratio gives it a distinct advantage if design on the basis of proof-stress such as BS 5500 is permissible, but even when designing to tensile-stress codes it is still a very economical material. It is readily welded, but fillers with the same composition as the parent metal must not be used as such welds lack adequate toughness. Austenitic 25% Cr-20% Ni consumables give tough welds with expansion coefficients that match those of the parent metal, but whose strengths are lower, whereas the higher-strength Inconel types have mismatching expansion coefficients and this can cause high contraction stresses to be set up during thermal cycling. Thus neither type of electrode is ideal and furthermore their high cost detracts somewhat from the favourable economics offered by the parent metal. It should be noted that 9% Ni steel, like other high-tensile steels, is particularly prone to hydrogen embrittlement and thus precautions have to be taken to prevent hydrogen pick-up during welding or in service. A special high quality version of 9% Ni steel was used for the fan shaft of the NTF. The possibility of using the standard alloy for the pressure shell must also have been considered seriously as it would have been a cost-effective alternative to the 304L stainless steel eventually chosen, the relative availabilities of the quantities and product forms required probably being one of the deciding factors.

5.2.2 Cryogenic Wind Tunnels with Ferritic Steel Pressure Shells

It should be noted that there are in fact a number of cryogenic wind tunnels in operation in which the pressure shell is made of mild steel and similar low alloy steels with tough-brittle transitions near room temperature. These may be divided into two categories, the first being blow-down tunnels or recirculating tunnels with short duty cycles. In these cases the tunnels are lined with a relatively thin layer of some form of durable insulation that can be given a clean aerodynamic profile. The thermal characteristics of the tunnel are analysed to establish that the thermal diffusivity of the insulation is low enough to prevent the walls from cooling significantly during the cold period of the tunnel duty cycle. The second type of tunnel is typified by the tunnel at Tsukuba University, Japan. Here the internal insulation is much thicker and the tunnel operates continuously. Thermocouples are used to sense the temperature of the insulation layer adjacent to the inner surface of the pressure shell and heater tapes are available to provide local heating should it be necessary to ensure that the wall temperature does not fall below its tough-brittle transition.

5.3 Fracture Toughness and Crack Propagation

In most materials the presence of a notch or flaw has an embrittling effect due to the stress concentrating effect at the tip. Modern theories of Linear Elastic and General Yieding Fracture Mechanics (LEFM) and (GYFM) can relate the fracture strength and critical flaw size to the Fracture Toughness of the material, Kc. In a simplified form the basic relationship is:

$$\sigma_f = K_c / [\pi a + 0.5(K_c/\sigma_y)^2]^{1/2}$$

where σ_f is the fracture stress, K_c the fracture toughness, σ_y is the yield stress and a is the critical crack length. Note particularly the term $(K_c/\sigma_y)^2$, as it gives an indication of the amount of plastic deformation that takes place in the material ahead of the advancing crack. If σ_y is small, (K_c/σ_y) is large and there is a large plastic zone ahead of the crack tip. Relatively large amounts of energy are absorbed in tearing through this zone and crack propagation is therefore made more difficult. In contrast, if σ_y is large, (K_c/σ_y) is relatively small, the plastic zone size is also small and little energy is absorbed by shear deformation. Furthermore, we have already seen that the yield stresses of many alloys increase quite rapidly as the temperature falls. Thus to maintain the same relationship between fracture strength and critical crack size the fracture toughness would have to increase in proportion to the yield stress. This does not happen in many high strength alloys and as a result the critical crack size decreases and they become increasingly notch brittle. Unfortunately the fracture toughness of a material is not only highly dependent on its physical and mechanical condition but also on its thickness and even sample width. Nevertheless the application of the concepts of fracture mechanics have provided a better understanding of fracture behaviour in high strength alloys and a firmer basis for design to prevent low energy absorbent fracture.

It is convenient to divide materials into the value of the ratio of the tensile modulus to the yield strength, E/σ_y. If E/σ_y is less than 150 the material has such a high strength that critical flaw sizes are low and load-bearing structures must be designed using fracture toughness analyses. If E/σ_y is greater than 150 the material is low strength and only those bcc metals such as ferritic steels that fail by cleavage lack toughness. Medium strength materials falling between these limits at room temperature can become effectively high strength materials at low temperatures because of the rise in their yield stresses. Fracture mechanics analyses are carried out for many of the high strength materials used in various cryogenic wind tunnel applications including the support stings, balances, models and other components that are highly stressed at cryogenic temperatures. Fracture toughness is considered further in the next lecture and selected values for Kc at 300 and 77K are given in its Appendix for many of the alloys likely to be of use for these applications.

5.4 Time-Dependent Failure

As has been shown, the fracture stress of a material is strongly influenced by the presence of cracks and flaws. There are three principle mechanisms by which such cracks may form or intensify during service: (1) fatigue, (2) corrosion (especially-stress corrosion and corrosion-fatigue), and (3) hydrogen embrittlement. None of these is a specifically low-temperature phenomenon, indeed the fatigue lives of many metals increase considerably at low temperatures, while the rates at which most corrosion reactions take place drop rapidly as the temperature falls - rather they increase the probability of unstable failure under service conditions which would normally be considered satisfactory. Their effect is the result of one or more of the following factors: (a) they lower the toughness of the material, (b) they provide a mechanism whereby a crack sharpens and increases the degree of stress concentration, or (c) they allow a sub critical crack to grow at stresses below the gross yield stress until it reaches the critical length required for unstable propagation.

5.4.1 Fatigue and Thermal Fatigue

Fatigue failure occurs in materials subjected to cyclic or fluctuating stresses which may or may not be superimposed on static applied stresses. Failure under such loading conditions can take place at stresses which are very much lower than the tensile or yield stresses even in materials which are normally considered to be tough and ductile. Fatigue must therefore be considered as a possible mode of failure in any piece of low-temperature equipment subjected to cyclic loading or vibration (for example, pumps, motors and turbines), or to periodic changes in pressure (transfer lines, storage vessels, and other process plant). The severity of the problem may be indicated by the estimate that about half the failures encountered in general engineering practice are caused by fatigue. Fatigue tests carried out at low temperatures have shown that fatigue lives of most metals rise considerably as the temperature falls. It has already been demonstrated that the tensile stresses of most metals increase as the temperature is lowered and it has been found that there is a strong correlation between fatigue strength and tensile strength; indeed, experiments have shown that in some metals the ratio of the endurance limit at 10^5 cycles to the tensile stress is virtually independent of testing temperature.

Thermal fatigue is of particular relevance to cryogenic plant, such failures having occurred in regenerators after a large number of temperature reversals, and in heat exchangers and other components after a relatively small number of warming and cooling cycles caused by plant shut-down. The basic cause of this type of fatigue is the high stresses and strains that can be set up during thermal cycling if temperature gradients are non-linear or if free expansion and contraction are restricted by external constraints. In this type of low-cycle, high-strain fatigue, small amounts of plastic deformation take place during each loading cycle and cumulatively lead to failure. Such failures were in fact encountered in the early operation of NASA LaRC 0.3-m TCT where "spoke-like" aluminium struts were rigidly attached to the tunnel pressure vessel and to central "hub-like" structures. Subsequent redesign using PTFE cushioned "T" slots at the central hub attachment points allowed free thermal expansion of the spokes and elimination of the stresses that caused thermal fatigue.

Pre-existing flaws, notches, cracks, badly radiused corners and other surface defects have a strong influence on fatigue lives, causing them to drop sharply at all temperatures even in materials which are not normally considered to be notch sensitive. This is usually a result of the increase in stress intensity brought about by the sharp fatigue cracks and it is particularly severe in high-strength aluminium alloys, titanium, and stainless steels which are known to be notch sensitive. As noted earlier, it is possible to apply the concepts of fracture toughness to crack growth under cyclic applied stresses and many data compilations (e.g. Ref. 18) show the relationship between crack growth rate da/dN as a function of stress intensity factor range ΔK. Predicted component lives are then obtained by calculating how many cycles are required to increase the flaw size from its initial to the critical value. In general, fatigue crack growth rates decrease at low temperatures for most metals normally used for the construction of cryogenic equipment. In contrast they increase for ferritic steels at temperatures below the ductile-brittle transition.

Corrosion fatigue can be a problem with some low temperature equipment particularly if it is frequently cycled between low and ambient temperatures. In the presence of even mildly corrosive environments large reductions in the endurance limits can occur even though the amount of metal corroded is negligible. Some aluminium alloys are especially prone to corrosion-fatigue, ordinary moist air causing some deterioration, while salt-laden atmospheres are particularly harmful. Premature failure has been known to occur in air separation plants located by the sea or near chemical plants and if such conditions are liable to be encountered it is necessary to apply a protective coating to the metal or to specify a material such as stainless steel, which is less susceptible to this type of failure.

5.4.2 Corrosion and Embrittlement

These are two mechanisms by which failure can occur without warning long after the initial application of the stress and they can cause failure at low temperatures even though the actual corrosion or embrittlement is more likely to have taken place at or above room temperature. Stress-corrosion resulting from internal residual stresses is liable to occur in brass, aluminium, magnesium, titanium, and steel as well as some non-metals. It is usually prevented by annealing at a temperature high enough to remove the residual stresses without weakening the material. Hydrogen embrittlement is a particular problem in high-strength steels such as 9% Ni steel. The hydrogen is usually absorbed during pickling and plating processes or during welding, and, although the hydrogen can sometimes be removed from steels by baking at 350 C, it is better to prevent its initial pick-up where possible.

6. STRENGTH AND TOUGHNESS OF NON-METALS AT LOW TEMPERATURES

Non-metallic materials have much more complex structures than metals and the amorphous and microcrystalline structures typically found in glasses and ceramics almost invariably make them brittle because they are unable to accommodate the plastic deformation needed to relieve the high stress concentrations which build up around small flaws. They are thus much stronger in compression than in tension but are rarely used in the bulk form even under compressive loadings because their poor thermal conductivities and consequent liability to thermal shock make them liable to shatter. However, in the finely divided form of fibres, powders, films, foams and expanded granules they are widely used for thermal and electrical insulation at low temperatures.

6.1 Ceramics and Glasses

Glasses and ceramics have amorphous structures and they are unable to deform plastically and relieve stress concentrations caused by microcracks in their surfaces. They fail in tension at relatively low stresses at all temperatures and their use in bulk form at low temperatures is limited. Glass Dewar flasks are, however, still used for storing small quantities of cryogenic fluids and large plate glass windows are employed as viewing ports in cryogenic bubble chambers. Careful thermal annealing to remove surface cracks and residual stresses is essential for these applications, while thick sections have to be cooled extremely slowly to avoid failure due to differential contraction or thermal shock. Ceramics and glasses are stronger in compression than in tension because the microcracks are propagated by tensile stresses and residual compressive stresses are often induced in the surfaces of glass plates to toughen them. In a similar manner concrete structures, which are also brittle in tension, can operate satisfactorily at low temperatures if kept in compression and large liquid-natural-gas storage tanks have been constructed in which the concrete is maintained in compression by steel reinforcing rods placed in tension around the warmer outside of the tank. The liquid nitrogen storage tanks for the University of Tsukuba cryogenic wind tunnel are also made of concrete lined internally to minimise the evaporation rate and keep the tension rods in the warm part of the concrete. For applications such as the floors of loading bays for road tankers transporting cryogenic liquids, it has been found that high-alumina cements such as Ciment Fondue are much more resistant to shattering by thermal shock than is ordinary Portland cement.

6.2 Thermoplastics and Thermosets

Polymeric materials can be divided basically into two structural categories: the thermoplastics and the thermosets. The long chain molecular structures of thermo-plastics give them mechanical properties which are strongly dependent on the temperature and rate at which they are stressed. Furthermore, an increase in their intermolecular forces over a temperature range known as the glass transition, which may be above or below ambient, means that most thermoplastics undergo a reversible transition to a glass-brittle state in which they are unable to deform plastically. As their glass transition temperatures are all above 150 K there are no thermoplastics which exhibit any really significant degree of ductility below this temperature.

Thermosetting polymers have a network structure which renders them brittle and they are thus rarely used in the unfilled st . However, when combined with suitable fillers their toughness is greatly improved and phenolic-impregnated cloths and papers (such as Tufnol and Paxolin) are particularly useful for the fabrication of load-bearing, electrically-insulating fitments. The inclusion of powdered glass or ceramic powders reduces the brittleness of un-filled resins while the incorporation of glass or carbon fibres and cloths in epoxy resin matrices gives the high performance composites to be considered in section 6.4.

Materials selection becomes even more complicated if these materials are needed in applications where liquid oxygen (LOX) is present, as virtually all hydrocarbon-based polymers are incompatible with LOX and burn violently in its presence. The polysulphides, silicones and fluorosilicones are more LOX compatible, but it is only the fluorocarbons that are completely satisfactory in this respect.

6.2.1 Thermoplastics of Particular Interest for Cryogenic Wind Tunnels

Applications of major interest for wind tunnel models will be considered in the next lecture. Here we will consider thermoplastics in their roles as seals and adhesives. Correctly designed fittings are important if leak-tight joints are to be made at low temperatures. As long as no dynamic stresses are involved, satisfactory seals can be obtained from elastomers, even when they are below their glass transition. In order to achieve this aim, very large compressive strains are imposed at room temperature so that the elastomer is able to exert sufficient force to offset the decrease in load caused by the contraction which takes place as it goes through its glass transition. The most satisfactory results are obtained using confined compression designs in which a follower on one flange squeezes the O-ring into the bottom of a matching groove in the face of the other flange, as well as into the clearance space between groove and follower. The O rings are typically compressed by about 80% of their original diameter and are thus not re-useable.

The fluorocarbon family of polymers, polytetrafluoroethylene (PTFE, TFE, Teflon), the fluorinated ethylene/propylene copolymers (FEP), and polychlorotrifluoroethylene (PCTFE F), are the only materials which retain any measurable ductility (approx. 1%) down to 4 K. This is a res of their unique molecular structure in which crystallites are formed having a tight spiral formation of fluorine and chlorine groups which are unable to pack closely together, thus preventing the material from having a strong glass transition. Although these materials are not elastomers, their ability to undergo enough plastic deformation to form a satisfactory seal makes them invaluable for use at temperatures down to 4K. They do, however, suffer from a tendency to cold flow under continuous load, and seals are thus liable to leak unless the load can be increased to compensate for the cold flow. Filled-PTFE compositions have been developed to overcome this problem, glass-fibre being most commonly used for O-rings and gaskets, while graphite, bronze and other powders are also used for bearing applications. Glass fibre improves the tensile and compressive properties of the materials, while the PTFE gives it sufficient ductility to accommodate plastically the strains developed. Furthermore, the thermal contraction of the composite is reduced from the high value characteristic of unfilled PTFE, so that the expansion of the composite is more compatible with that of metals, thus making joint design easier. The confined compression designs of flanges are also to be preferred for use with fluorocarbon seals because they minimise the deleterious effects of cold flow.

In the NASA LaRC 0.3-m TCT many large and small diameter seals are made using "Gortex". This is a low-density form of PTFE created by a proprietary process that involves stretching the material to about 10 times its original length without reducing its diameter. The material has been found to be particularly suitable for the demountable seals on the plenum cover of the 2D test section. Although "Gortex" is not intended for re-use, it has been found that it is not necessary to replace the gaskets each time the cover is opened. An alternative approach is to use spring- or pressure-assisted seals. In spring-assisted seals a metal backbone spring provides the sealing force and helps to compensate for dimensional changes during cool-down, while the PTFE coating forms the actual seal with the mating surface. A "U" shape cross section seal with a type 302 stainless steel spring and PTFE coating was chosen for the NTF, the "U" shape section allowing the use of gas pressure to improve sealing efficiency. It was also found that the surface finish of the mating surface was important, a 0.8 μm (32 μin) RMS finish being acceptable, and that treatment of these surfaces with FEP or TFE tapes or lubricants improved sealing efficiency.

There are many applications in which metal-metal, metal-plastics or plastic-plastics adhesive joints are advantageous. For example, brackets, clips and other attachments can be bonded to pressure vessels without creating the stress raisers that would otherwise be caused by welding or other conventional techniques, while corrosion-free joints between dissimilar metals can be obtained if their surfaces are kept apart by an electrically non-conducting adhesive. At room temperature, adhesives are usually able to deform sufficiently for any stress concentrations to be relieved, but at low temperatures their moduli increase considerably and make this much less likely. Contraction and other stresses have to be minimized

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and the key to the successful development of structural adhesives lies in the use of fillers that match the expansion coefficients of the adhesives as closely as possible to those of the substrate and the adherent. They also redistribute thermal stresses throughout the adhesive instead of concentrating them at the adhesive-substrate interface. Even so, the thermal conductivities of most adhesives are low, and temperature differentials between them and metal substrates can cause failure from thermal shock if the glue line is not kept as thin as possible. One way of achieving this is to use a 'structure' or 'carrier' between adherent and substrate. This is usually a thin layer of glass fibre mat which allows the adhesive to penetrate and wet the filaments, thus forming an even bond as well as reducing the differential contraction between adhesive and adherent. It also has the further advantage of reducing creep at ambient temperature in adhesives such as the polyurethane pastes, which in other respects are among the most successful adhesives for low-temperature applications. Other types include the epoxy-nylons, nitrile modified phenolics, epoxy-phenolics, and fluorocarbon-epoxy-polyamides.

6.3 High-Performance Composites

High performance composites are formed by the incorporation of glass, carbon or Kevlar fibres in matrices of thermosetting polymers such as epoxy resins. For most aerospace applications their attraction lies in their high specific strengths and stiffness, and these characteristics improve at low temperatures but not, perhaps, so markedly as those of metals. One major interest in glass-reinforced plastics for low temperature applications comes, as noted earlier, from their very high strength/thermal conductivity ratios which make them ideal for use as load-bearing, thermally-insulating supports.

As the reinforcing fibres are stronger and stiffer than the matrix, they support the greater part of the applied load and the strength of the composite is determined by the length, orientation, and concentration of the fibres. 50-60 per cent of fibres by volume are typical maximum concentrations unless filament-winding techniques are used. Tensile strengths of 270-420 MPa are typical of room-temperature values and these increase gradually to about 480-700 MPa at 77 K. Their moduli are less temperature dependent, increasing by about 10-20 per cent on cooling from 300 K to 20 K, while their toughness, as measured by impact or notched tensile tests, shows little significant variation over this temperature range. These are quite high strengths by most standards, and when the low density of glass-fibre-reinforced plastics (GRP) is taken into consideration, it can be seen that their specific strengths are extremely high.

Tensile tests carried out on specimens laminated from a single thickness of woven cloth show a change from a high initial modulus to a lower secondary modulus at a load equivalent to about 13-15 per cent of their ultimate load-carrying capacities. If the specimens are examined at this stage they can be seen to be full of microcracks and the material is now porous and unable to retain vapour or liquids. The root of this difficulty lies in the failure of the bond at the fibre-matrix interface in those fibres that have a large stress component resolved perpendicular to the fibres. The strength of this bond is about 12-15 per cent of the composite strength parallel to the fibres. Hence the material has become porous long before it has developed its full potential strength and its electrical properties are also degraded. Fatigue failure also develops by fibre/matrix debonding and resin cracking at stresses lower than those in comparable static tests. Where fatigue loading is expected, design stresses are usually taken as about one-tenth of the composite failure stress.

Glass fibre reinforced composites also have other drawbacks. Static fatigue, which is a characteristic failure mode in bulk glass and unreinforced thermo-plastics, can also occur in GRPs if moisture is able to penetrate the fibre matrix interface, although this failure mechanism does not operate if the composite is maintained at low temperatures. A more serious difficulty lies in the highly anisotropic nature of their mechanical properties, reinforcement being much more efficient in a direction parallel to the fibres than perpendicular to them. Cross-plying the lamination allows two-dimensional reinforcement, but strengths and moduli are reduced compared to those attainable parallel to the fibres. If the orientation of successive plies is varied from layer to layer, this anisotropy can be reduced and more homogeneous properties obtained. This type of laminated structure was utilized in the construction of the NTF fan blades. Two different types of woven E glass cloth pre-impregnated with an epoxy resin were laid up at predetermined orientations and oven cured to consolidate the laminate and fully cure the resin. Other fibreglass structures used in the NTF include pultrusion sections used to fix the foamed glass insulation between the inner aluminium liner and outer stainless steel pressure shell. Further applications of high performance composites will be considered in the next lecture.

7. MATERIAL PROCUREMENT AND QUALITY CONTROL

It should be noted that the construction of cryogenic equipment is subject to the same economic constraints as any other large technical project, in that the total cost of each stage or component needs to be considered when alternative materials are being considered. Thus, to the cost of the basic material needs to be added the cost of the appropriate forming, joining and fabrication processes, inspection and quality control, possible rework and final finishing. It is almost invariably a false economy to purchase material for demanding technical applications at 'rock-bottom' prices as the resultant 'savings' frequently lead to subsequent costly problems, or even rejection of the component. The additional costs incurred in ensuring that a project starts off with top quality material are a worthwhile premium to pay for avoiding the problems likely to arise from the use of poor quality material. It is recommended that the ultimate use of component, and possibly the intended fabrication route should be made known to the materials suppliers when quotations are being sought so that they are aware of the problems that might arise if target specifications are not met. Furthermore, it has often been found that 'misunderstandings' are kept to a minimum if the project engineer makes contact with a technical representative of the suppliers to make him aware of the project requirements, rather than just leaving the purchasing department to progress the order.

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8. CONCLUSIONS

In this lecture the author has tried to bring out the philosophy that cryogenic engineering is a mature technology that has much to offer the field of aerodynamic testing through use of the cryogenic wind tunnel. It is, however, important that those without a thorough grounding in cryogenic engineering should make an effort to beflfit from accumulated experience on the correct way to handle cryogenic fluids and the best materials to us in the construction of equipment for operation at cryogenic temperatures. To this end, particular emphasis has been laid in this lecture on those aspects of cryogenic engineering that have a direct bearing on safety, in the belief that if potential problems can be understood, it is less likely that actual problems will be encountered. Used with care cryogenic fluids such as liquid nitrogen can bring significant scientific and technical advantages. Misused, the cost could be injury, or even death, of those directly or indirectly involved.

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10. ACKNOWLEDGEMENTS

The author would like to thank all those who have assisted in the preparation of this manuscript, particularly Dr Robert Kilgore of NASA Langley Research Center who helped to determine its scope and contents, Mrs Peggy McGuire who spent many hours reading the proofs and Jackie Halliday and Carolyn Wigley for much of the typing.

1. Report No.
NASA CR-177932

2. Government Accession No.

3. Recipient's Catalog No.

4. Title and Subtitle
Basic Cryogenics and Materials

5. Report Date
June 1985

6. Performing Organization Code

7. Author(s)
David A. Wigley

8. Performing Organization Report No.

9. Performing Organization Name and Address
Applied Cryogenics and Materials Consultants, Inc.
Basin Road Industrial Center
P. O. Box 765
New Castle, DE 19720

10. Work Unit No.

11. Contract or Grant No.
NAS1-17571

12. Sponsoring Agency Name and Address
National Aeronautics and Space Administration
Washington, D.C. 20546

13. Type of Report and Period Covered
Contractor Report

14. Sponsoring Agency Code
324-01-00

15. Supplementary Notes
Langley Technical Monitor: Pierce L. Lawing
Small Business Innovative Research Program (SBIR)
Phase I, Supplemental Report

16. Abstract
This paper summarizes the effects of cryogenic temperatures on the mechanical and physical properties of materials. Heat capacity and thermal conductivity are considered in the context of conservation of liquid nitrogen, thermal stability of the gas stream and the response time for changes in operating temperature. Particular attention is given to the effects of differential expansion and failure due to thermal fatigue. Factors affecting safety are discussed, including hazards created due to the inadvertent production of liquid oxygen and the physiological effects of exposure to liquid and gaseous nitrogen, such as cold burns and asphyxiation. The preference for using f.c.c metals at low temperatures is explained in terms of their superior toughness and the limitations on the use of ferritic steels is also considered. Non-metallic materials are discussed, mainly in the context of their LOX compatibility and their use in the form of foams and fibres as insultants, seals and fibre-reinforced composites.

17. Key Words (Suggested by Author(s)
Cryogenics
Materials

18. Distribution Statement
Unclassified - Unlimited
Subject Categories 09, 26, 27, and 31

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price
Unclassified	Unclassified	19	A02

For sale by the National Technical Information Service, Springfield, Virginia 22161