Characterisation of high-temperature materials

MECHANICAL TESTING

edited by I Curbishley

The Institute of Metals

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PREFACE

This is the third in a series of seven monographs presenting a broad practical overview of modern methods of characterising high-temperature materials. It follows the publications on microstructural and chemical characterisation. Later volumes in the series will cover physical and elastic characterisation, non-destructive testing, surface stability (including corrosion and tribology) and numerical techniques. Although the series is primarily concerned with techniques used for characterising the properties of materials used at high temperatures, many of the techniques are also more generally applicable to materials used at any temperature.

This volume covers tensile testing, creep, fatigue crack growth, both low cycle (high strain) fatigue and high cycle fatigue, and fracture toughness testing. Many of these areas have developed greatly within recent years, and the methods of testing and analysis are still being actively developed. The contributors to this volume have been closely involved with the developments in their respective areas.

The aim throughout is to allow the non-specialist to appreciate what types of test are available, to help select the most appropriate for his requirements, and also to appreciate the manner in which published data to which he may refer have been produced and analysed for presentation. With the current emphasis throughout industry on Quality Assurance there is much interest at present in testing methods, including the development of British and International Standards. The authors have also aimed therefore to give guidance on the developments in these Standards.

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1 INTRODUCTION

1.1 Function Of Tensile Testing

The tensile test is the most obvious kind of mechanical test which can be carried out on a material and it is therefore applied more often than most others. Indeed, many of the other forms of mechanical test related to fatigue, creep or notch toughness for example, were only developed in each case when it became obvious that simple tensile test results were inadequate to characterise the particular behaviour. Tensile tests are multi-purpose in character, in the sense that the results are typically applied in a variety of contexts. For example, the purpose may be to:

- obtain data relevant to design or the prediction of service performance
- provide indices which will be used to compare materials for selection purposes
- provide information for quality control
- obtain data relevant to the control of forming or fabrication processes
- provide a tool for fundamental studies of material behaviour

These different applications often become confused and ideally, different procedures and measurements should be employed depending on which function the tensile test is supposed to be fulfilling. For example, for quality control, the main emphasis might be on comparability and convenience, whereas tests related to service performance should be more concerned with accurate simulation of the service environment. For some materials, different standard procedures, appropriate to different functions, have in fact been established, but for most materials, only one standard is available to cover all needs.

1.2 High-Temperature Design Parameters

Matching the test procedure to the function is particularly problematic for high temperature materials as the behaviour of the material may be time-dependent and it is often unrealistic to expect simulation of real time-scales. There are of course some high-temperature applications where a short-term tensile test result will be directly useful; for example, in high-temperature forming studies or for predictions relating to accidental over-temperature excursions. However, in most normal applications of high-temperature materials, interaction of time and temperature must be considered in deciding on the relevance of a given test procedure to given design conditions.

Practical design procedures for metals are based on the idea that various failure modes can be avoided by indexing the design stress to the lowest of three characteristic stress levels related to:

- short-term tensile properties (eg **yield** and **tensile strength**)
- stress to produce an arbitrary **creep deformation** (eg 1%) in the design life (eg 100,000 hours)
- stress to **rupture** the material in the design life

Fig. 1 shows in a schematic manner how these alternative criteria could relate for a typical high-temperature material subject to steady loading. Below a certain temperature, which depends very much on arbitrary limits set for creep deformation and life, time-independence is assumed and simple tensile properties may be utilised. This therefore defines a temperature regime in which short-term tensile tests give useful information; creep deformation may still occur in this regime but the creep rates at the chosen design stress level are hopefully insignificant. Hence, essentially elastic design criteria can be invoked.

Above this temperature, design should be based on creep criteria and there have been many attempts over the years to validate approaches whereby short-term creep tests (albeit of a longer duration than normal tensile tests) could be used to predict long-term creep properties. However such tests are not regarded as being sufficiently reliable, even for the purposes of material selection.[1] The distinction between 'short-term' tensile testing and other time-scales is in any case an arbitrary one and is determined largely by the equipment used. For the present purposes, a similar classification to that used by Gillis and Gross is adopted,[2] where different forms of test are identified by the nominal strain rates employed:

Test Type	Strain Rate (min^{-1})
creep	10^{-8} to 10^{-4}
short-term tensile	10^{-4} to 10
dynamic and impact	10 to 10^{+5}

2 DEVELOPMENT OF TENSILE TESTING PRACTICE

2.1 Introduction

Tensile testing has always occupied a central role in the design of load-carrying structures and strength technology in general. Interest in the *high-temperature* capability of materials was related in the first instance to the development of heat engines in the early 19th Century and William Fairbairn is credited with the first application of tensile tests at high temperature in Britain. He carried out his tests by transferring standard specimens without delay from a soak furnace to a conventional testing machine. He found that '*at red heat*' the strength of wrought iron was reduced to 5.0 tons/in^2, when it had been 27.6 tons/in^2 at ambient temperature. Methods for measurement of temperature were obviously rather underdeveloped in those days, but these test results were nevertheless entirely useful for service situations where the control of temperature was equally uncertain.

However, although the high-temperature aspect of Fairbairn's tests was novel, his equipment was based on an extensive body of experience in ambient temperature tensile testing, dating back to Leonardo da Vinci's famous tests on iron wire. Some of this experience is worth recounting, as it forms the background to modern testing technology. The simplicity of approach typical of earlier times is also worth recovering. The aim of the following sections therefore is to highlight some of the important issues in tensile testing through a selective account of various landmarks in the historical development of the technology. This treatment draws, among other sources, on Stephen Timoshenko's definitive 'History of Strength of Materials'.[3]

2.2 On Sampling and Similarity

Galileo (1564-1642) was the first to propose *cross-sectional area* as a characterising parameter for strength calculations and tests. He showed how to relate the failure strengths of small samples and full-scale structures (Fig. 2). However the need to scale other dimensions of a test piece aside from the cross-sectional area was not clearly established until the late 19th Century and it is worth recalling the background to this discovery.

By the middle of the 19th Century, more and more iron and steel products were appearing on the market and users of these products turned to tensile testing to settle rival claims about strength and performance. The Glasgow engine makers, Robert Napier and Sons, commissioned David Kirkaldy to set up a testing machine (Fig. 3) which would be used to assess the relative merits of two potential replacements for wrought iron, designated *'puddle steel'* and *'homogeneous metal'*. His machine may appear crude, even by standards then current, but Kirkaldy applied an enquiring mind and acute observation to produce, according to Timoshenko, *'the most complete description of the mechanical properties of iron and steel then available'*.[4] Kirkaldy was the first to suggest that reduction-in-area at failure could be used to characterise ductility and thus drew attention to the need to balance high strength with adequate ductility. He also observed various effects of specimen shape and dimensions on strength and ductility and stated clearly for the first time that the failure stress of a given material could not simply be assumed to be an invariant property of the material; it depended greatly on test procedures and specimen geometry. During the present upsurge of activity related to the harmonisation of standards in Europe, it is worth recalling his words emphasising *'the absolute necessity of correctly knowing the exact conditions under which any tests are made before we can equitably compare results from different quarters'*.

By the end of the 19th Century, several meetings to establish uniformity of technique in mechanical testing had been held in European centres and the importance of employing specimens which would be *geometrically similar* in all respects came to be understood. The application of this principle in relation to ductile failure-elongation is attributed to J. Barba, who stated that the same percentage elongations are obtained in geometrically-scaled cylindrical specimens when the index gauge lengths are corresponding

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multiples of the specimen diameter. The same conclusion appears to hold approximately true for gauge-length-to-thickness multiples in the case of rectangular cross-sections of a given width-to-thickness ratio.[5]

Interpretation of Barba's Law is more difficult when the specimen geometries to be compared are not strictly similar. In comparing results from tests on rectangular cross-sections of varying aspect ratio, there is some experimental support for the notion that the square root of cross-sectional area is an appropriate normalising parameter; that is, the same percentage elongation is obtained using a gauge length which is a given multiple of the square root of cross-sectional area (see ref. 5, pp 102-107). However this conclusion should not be carried to extremes, for example to expect a valid comparison between square bars and thin sheet, or between circular and rectangular cross-sections. The triaxial constraints which develop on plastic necks could be entirely different in such cases, leading to different apparent properties.

Once it had been accepted that a dimensionally similar gauge length should be used, various opinions developed concerning the precise choice of gauge length/diameter multiple. National Standards in different countries in the earlier part of the present century called for gauge lengths varying between 3.5 x diameter and 10 x diameter. The factor which complicates the decision is that the total plastic elongation at failure usually consists of two distinct parts; a uniform parallel elongation and a non-uniform or 'necked' portion. If a short gauge length is specified, this emphasises the necking process, whereas a very large gauge length tends to de-emphasise it. As the neck is commonly found to be somewhere between half-a-diameter and two diameters long, depending on material, a gauge length somewhat greater than two diameters should provide reasonable insensitivity to necking variations, as shown in Fig. 4 (data from Davis *et al*).[6] Currently, the most commonly accepted gauge length in the case of cylindrical specimens is 5 diameters - equivalent to 5.65 x √Area; (4 x d in the US).

There are, however some significant exceptions to the principle that the behaviour of tensile specimens can be normalised on the basis of dimensional similarity. These mostly relate to the fact that real materials are not always homogeneous. For example, one of the intriguing aspects of Leonardo's test results on iron wire was that the strength of long wires was less than for short wires. The reason seems to have been that, for the metallurgical techniques then available, the likelihood of a critical defect being present increased with the length of wire sample. A more precise understanding of failure by defect propagation was not provided until the formulation of fracture mechanics, when it became apparent that geometrical scaling of crack size, in step with other dimensions, does not produce identical failure stress. Until this was understood, service failures and practical tests which appeared to contradict Barba's Law were often treated with great scepticism despite the compelling evidence from many sources that larger, thicker structures had a greater tendency to catastrophic failure.

Even when significant defects are absent, extrapolation of small-specimen behaviour can be a dubious exercise if the material exhibits point-to-point variations. This is a particularly common trap in relation to the use of testing machines which have only a modest load capacity. To give an example - many texts identify the Dutch physicist Petrus van Musschenbroek (1692-1791) as the father of the tensile testing machine (Fig. 5) and his design certainly provided a degree of control of loading which had not previously been available. However the penalty was that the specimens had to be small, as the machine was modest in capacity. Van Musschenbroek's work was therefore criticised at a later time by the French engineer Buffon (1707-1788) on the grounds that he had drawn misleading conclusions about the failure of full-scale timber structures from the results of tests on small samples of wood. Buffon himself showed that the strength of wood samples depended greatly on the point in the tree trunk they were drawn from and thus he demonstrated the need for machines which would be powerful enough to load representative sections of material. The validity of this argument is obvious enough for timber but it is equally significant for plastics, metals and ceramics which frequently exhibit spatial variations related to processing or composition. The principle of adopting the largest specimen size possible, together with the biggest load-capacity machine available, remains a good one.

2.3 On Stressing ....

The single-lever or 'steelyard' principle adopted by Musschenbroek is still in use today, despite its technical limitations. The main advantage of this design was that it provided a virtually stepless increase in applied load. The French engineer Rondelet (1794-1829) improved the basic design by substituting knife-edge pivots to reduce friction and this feature has been in use ever since in single and multi-lever loading and weighing systems.

The need which arose in the mid-19th Century to test larger specimens and more ductile materials, exposed another weakness of the simple lever machine; if the specimen stretched a long way, the angle of the balance beam could change sufficiently to alter the applied load. One common solution to this problem, shown in Fig. 6, was to attach one end of the specimen to a hydraulic piston which could be pumped out as necessary to absorb the stretch and thereby allow the beam to be balanced in a level position. Machines using this principle, having capacities of 100 Tons or more, were constructed in the latter half of the 19th Century by Kirkaldy in England, Lamé in Russia and L. Werder in Germany. The Werder machine was adopted in most of the State testing laboratories in Germany and was the mainstay of much pioneering work in materials research. Screw mechanisms were also used to absorb plastic stretch but as these were operated manually, they were more common on lighter capacity machines.

The greater control offered by machines which were able to keep up with the specimen extension led to more accurate observations of post-yield phenomena; Lamé was the first to remark on the rapid stretching of iron samples at about two-thirds of the ultimate strength. However, as it was not easy in such machines to reduce the load during this phase, accurate observations of the yield drop were not reported until much later; C. Bach coined the terms '*upper and lower yield strength*' in 1902.

Lever machines required two simultaneous actions by the operator(s); the first to follow the stretch and the second to balance the weight system. This was obviously inconvenient. Machines designed in the present century began to feature '*self-indication*', whereby the force on the specimen was reacted by a pendulum weight or a spring, through a single or compound lever system.[7] The load-weighing mechanism would then always be in static balance and the pendulum or spring could be made to drive a pointer or other indicating device.

Other load indicating systems have been based on hydraulics - indeed it seems a simple and obvious idea to add a pressure gauge to an existing hydraulic ram system to indicate load. However, in normal rams, the variation in seal friction makes it very difficult to achieve sufficient accuracy. The solution to this problem was to eliminate the seal or packing and rely on a constantly running pump to compensate for the resulting leakage past the piston. The pressure registered in the cylinder could then be used to drive a variety of accurate self-indicating devices. Another approach, popular in the United States and described in ref.6, made use of a separate sealed capsule of hydraulic fluid to react the test load (the original 'load cell').

All of these load-indicating systems suffer the same drawback, namely that they are essentially quasi-static devices, and the development in modern times of load cells which feature minimal moving parts, has established a capability for accurate dynamic load measurement which has never before been available.

2.4 .... and Straining

The main preoccupation in early testing was with the load or stress at failure. Strain measurements were not made except by Hooke and Hodgkinson in relation to elasticity. They used very long (fifty foot) samples in order to generate large enough extensions to be measurable using dividers. The first sensitive measurements of strain on a normal, short tensile specimen were carried out by Johann Bauschinger (1833-1893) these deriving from his invention of the mirror extensometer. The use of reflected light beams to magnify the small extensions of the specimen in the elastic range was very effective as it avoided the inertia and lost-motion problems of mechanical lever-amplification systems. Strain was measured on both sides of the specimen so that bending could be detected and the instrument was capable of measuring an extension as small as 10^{-4} mm. Many variants of this system have been invented and some are still in use. Elevated-temperature testing has also depended on mirror-extensometers, in which case the movements of the gauge points were transmitted via long rods of high-temperature material to a point outside the furnace, where the measuring elements could continue to operate in a cooler environment.

Bauschinger, who carried out his tests at Munich using a Werder machine, was the first to conclude that the elastic limit and the proportional limit coincide. He also discovered the effect, which bears his name, of reversing the straining direction. It remains true that many important effects on yielding related to heat-treatment, cold-working or residual stress cannot be revealed without sensitive measurement of strain.

Autographic recording (from Greek 'self writing') of load and extension has been known at least since 1877, when Abbott devised a chart recorder where the load axis was driven by a pendulum self-indicating device. Many other systems based on mechanically driven pens and photographic recorders have been invented since then. In some machines, the 'strain' axis is driven by the moving crosshead or grip and this means that the record includes the deformation or free movement of various parts of the machine along with the desired stretch of the specimen (see section 3.5).

The good feature of all such autographic recorders was that rate effects could be examined, as there was no need to interrupt progress to take a reading on a separate manual extensometer. This advantage is especially relevant to elevated-temperature testing where creep strain can develop during the variable time taken to make a determination. Autographic recorders can also *conceal* rate effects however; although the record relates extension and load at every instant, the rate of development of strain or stress is not revealed unless it is logged independently. Dynamic effects related to overshoot of the weighing system or to inertia in the strain measurement device, can also obscure the true material response.

Until recently, autographic systems were normally less accurate than manual extensometers but they nevertheless offered much clearer observation of phenomena associated with yielding and work-hardening. However, the added complication of strain measurement has never been willingly accepted in the context of routine testing and some methods used in practice for the detection of yield are little better than they were over a hundred years ago. These methods are named in certain testing standards as the '*drop-of-beam*' or '*drop-of-pointer*' method and they rely on the fact that if the straining rate is more or less constant, the balancing load required as the specimen begins to yield will drop momentarily, or at least *hesitate* in its upward progress. These methods have survived to the present day when many machines have neither 'beams' nor 'pointers' but indicate load by a digital display; in which case the interpretation of 'hesitation' seems somewhat dubious.

2.5 On Testing Rate

It has been known for at least a century that measured properties are affected by testing rate. However, until recently, it has been difficult to quantify the effects properly, because the control of testing rate in standard tensile machines was always rather uncertain; particularly in the case of manually-balanced lever machines. In the case of ductile materials, the apparent trend is for yield strength and ultimate strength to increase with testing rate, especially at elevated temperature, but there were many conflicting indications with respect to ductility. Most researchers found that they had to build special machines to explore the effects.

Two approaches to rate control can be seen in these special studies. In the first, a constant **loading** rate was imposed on the specimen and in the second a constant **extension** rate was applied. Professor A. Barr devised a small autographic wire testing machine according to the constant loading rate principle and used it to illustrate a lecture to the West of Scotland Iron and Steel Institute in 1908 (Fig. 7).[8] The constant rate was obtained by running sand into the load pan in a steady flow and the extension was measured directly on a very large gauge length. The strain rate in such a machine goes up rather sharply on yielding, as the machine is unable to unload, and the effects on yield and ultimate strength were very marked. The testing rate on this machine therefore represented an upper bound on the rate of loading which would be produced by a lever machine where the operator moves the balance weight steadily along the beam without pause. Barr's tests explored a variety of effects, including work hardening and heat treatment and they led him to conclude that *'the apparent mechanical properties of materials depend greatly on their prior treatment and manner of testing'*. It seemed to him, therefore to be *'rather ridiculous to quote yield strength to several decimal places.'*

Tests using an arrangement which seems at first glance to represent the opposite extreme of constant extension rate, were reported by J L M Morrison in 1934.[9] In this case the extensions were produced by a constant speed electric motor and gearing to give nominal strain rates in a range from 10^{-6} to 10 min^{-1}. An autographic recording system which made use of photographic film was built into the equipment to improve dynamic capability. Morrison noted that this constant speed arrangement did not in fact produce constant extension rate of the specimen throughout the test; during the elastic phase, a substantial proportion of the crosshead motion was absorbed by deflection of the machine frame and the loading train, whereas during the plastic phase, the deflection of the machine parts was negligible compared to the extension of the specimen (See also 3.5).

His results showed that all strength properties tended to increase according to the logarithm of strain rate, the ultimate strength being less sensitive than the upper and lower yield strength values. He remarked that, within the range of test times typical of routine testing, the variation in strength properties was quite large enough to put the test material outside the range of values implied by the manufacturer's specification. He suggested that such a variation should not be left to the discretion of the machine operator and that it pointed to the need for machines which would provide more accurate speed control, as speed alone could account for at least 10% difference in the results obtained on nominally similar machines.

3 PRINCIPLES OF MODERN TESTING MACHINES

3.1 Introduction

A wide variety of tensile testing equipment is commonly found in test houses and laboratories and it is important to understand how a given machine which is to be used actually works. DIN Standard 51 221 is one of the few standards to lay down certain requirements for testing machines, although some aspects such as load measurement (BS 1610) and extensometer accuracy (BS 3846) are covered by other UK, US and International standards. The important features

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