TENSILE TESTING OF IRRADIATED MINIATURE SPECIMENS

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ABSTRACT

The use of miniature specimens is stimulated in order to economize on irradiation time. Additional advantages of working with miniature specimens include reduced space requirements during reactor irradiation and reduced radio-activity after irradiation.

For a number of years different subsize or miniature specimens are tested at ECN. In **Figure 1** the various types are shown. This paper describes the experiences on miniature tensile specimens with emphasis on the instrumentation.

Miniature tensile specimens were succesfully used to investigate high temperature helium embrittlement of alloys. Helium was introduced in the specimens by α-particle irradiation in a cyclotron. Thickness of the specimens was limited to 0.25 mm by the range of the α-particles. The gauge length and width of the specimens, 8 and 1.5 mm respectively, were kept small to minimize the cyclotron irradiation time and, to a smaller extent, to keep the reactor volume small during subsequent neutron irradiation.

To load specimens in a testing machine by remote handling without deformation prior to the actual test turned out to be a challenging problem. A procedure which was developed to do so reliably, is described in this paper. Additional aspects, such as fabrication of specimens and reproducibility of the results, are discussed.

INTRODUCTION

Helium embrittlement experiments at ECN [1], whereby helium was introduced in the specimens by α-particle irradiation, stimulated use of miniature specimens in order to economize on cyclotron time. Additional advantages of working with miniature specimens include reduced space requirements during reactor irradiation and reduced radio-activity after irradiation.

Because time for development of a miniature tensile specimen, including verification using full size specimens, was lacking, our approach was to scan the literature [2,3,4,5] and adopt a specimen type with proven performance and dimensions suitable for our experiment.

In this paper experience with the adopted sheet-type specimen is described, including the procedure for loading the delicate specimen in a testing machine by remote handling.

SPECIMEN GEOMETRY

The geometry of the specimens used in these experiments, shown in **Figure 2**, is very similar to one of several specimens developed for irradiation damage studies in the USA in the early eighties [3]. A few minor dimensional changes, such as no reduced width in the gauge length section of the 25 mm long specimen, were made in order to facilitate fabrication.

Comparison of the various specimens developed in the USA, three flat tensile specimens with lengths of 25, 32, and 44 mm and a 39 mm cilindrical-type tensile specimen, all made of a single heat of stainless steel 316, was reported [3]. Tests were carried out at room temperature, 300, and 600°C. The tensile properties determined with these specimens were shown to be in acceptable agreement. One of the studies concerns a comparison study on vanadium alloy specimens.

SPECIMEN FABRICATION AND CONDITIONING

A low purity vanadium (99.8%V) and a V-20Ti alloy were investigated. The materials were obtained with a thickness of 1 mm. The required thickness of 0.25 mm was achieved by cold rolling. After each rolling step a vacuum heat treatment was given to obtain a solution annealed condition.
The low purity vanadium was cold rolled in two steps and vacuum annealed at 1000°C for 1 hour. The V-20Ti was cold rolled from 1 mm to 0.25 mm in one step and solution annealed in vacuum at 1100°C for 30 minutes. This treatment resulted in average grain sizes from 30 and 40 μm respectively.
Miniature specimens were machined to the dimensions as shown in **Figure 2** .

Before machining of the specimens, the material was inspected on imperfections such as rolled-in particles, scratches, pits, folds and bends, rolling direction and dimensions. Next, the strips were cut to approximately the width of the specimens, followed by cutting the strips to the length of the specimens. At this stage the specimens are over-sized by 0.1 mm. From the specimens packages were made to drill the holes. The shape and dimensions were achieved by fine mill cutting of the packages. After machining and deburring all specimens were coded. Finally, a dimension check was executed on a number of specimens. The mean width was 1.489 mm with a standard deviation of 2.7%, the mean thickness was 0.252 mm with a standard deviation of 3.3%. The mean gauge length was 7.973 mm with a standard deviation of 1.1%.

HELIUM IMPLANTATION

The cyclotron of the Joint Research Center (JRC), Ispra, Italy, was used to implant helium in specimens. The gauge section of a number of specimens of the alloys was irradiated with 36 MeV α-particles and an average beam current of 3-5μA to obtain a helium concentration of approximately 100 appm. A rotating aluminium disk of variable thickness intersecting the beam was used to implant helium uniformly over the specimen thickness. During irradiation the specimens were cooled by a flow of purified helium. The temperature of the specimens was approximately 50°C with maxima occurring up to 100°C. Actual helium concentrations still have to be determined. However, previous determinations confirmed the calculated concentrations to within 10%.

NEUTRON IRRADIATION

Specimen irradiation was performed at the High Flux Reactor (HFR) of the JRC-Petten, the Netherlands, to investigate both the helium implantation and displacement damage on the tensile properties of the materials. The irradiation experiment was performed in a SIENA capsule. The samples were loaded in three sample holders and irradiated at 600, 700, and 800°C, respectively. Each sample holder contained 70 miniature flat tensile specimens. A uniform temperature distribution over the length of the sample holders was obtained by using a mixture of helium/neon or argon gas. The capsule was irradiated during 7 HFR cycles up to 5 dpa.

TESTING FACILITY

The tests were performed on a small servohydraulic testing system, consisting of a plain bearing actuator with a static force of about 10 kN. This facility, shown in **Figure** 3, was also provided with a bell jar type vacuum chamber made of quartz, in which a vacuum of 10^{-5} Pa could be achieved.
The heating device consisted of a two zone lamp furnace and controlled by self-tuning temperature controllers. The temperature could be held to within +/-5°C in the range up to 800°C. Calibrated type K thermocouples were used to measure the temperature, which was recorded in an analogous way. The tests were started after a stabilisation period of 45 minutes. The tensile tests were performed at a constant strain rate of $10^{-4}.s^{-1}$. The tensile properties were obtained graphically from the load-displacement curves. The total elongation was determined graphically after fracture. No deformation was found outside the gauge section of the specimens.

REMOTE HANDLING OF SPECIMENS

The most complex in-cell operation was mounting of the specimen in the testing system. The main apparatus, schematically shown in **Figure 4**, was previously used to test subsize flat specimens, having a gauge length of 22 mm, a total length of 50 mm, and a cross-section of 3x1 mm.

To accommodate the miniature flat specimens in the existing loading train, adaptators were developed. It had to have the possibility for self-alignment to avoid bending in the gauge section of the specimen. Also, bending of the specimen during remote handling of the specimen and insertion in the loading train had to be avoided. For this reason a special assembly template was developed of which a schematic is given in **Figure 5**.

The two adaptors (1) are sunk down in the template and each provided with pins (2). The position is such that the slit of the adaptors are at the same level as the specimen centre line. After inserting the pins the adaptors are slid to each other until it is stopped by a centre pin (3) in the template. Then the specimen (4) is pushed in the slit of the adaptors and positioned by the same centre pin and provided with the connecting pins (5). The two adaptors with the mounted specimen is then carefully lifted from the template by the manipulator and put into the loading train. Loading is performed through the respective pins (5).

The specimen is mounted in such a way that it cannot bend during placement in the loading train. In addition, during the heating sequence a good temperature gradient of the specimen is then achieved.

CONCLUSIONS

The specimen described in this paper has been used to determine the effect of helium and neutron irradiation on the tensile properties of vanadium and vanadium alloys.

The procedure described to perform tensile testing by remote handling using miniature specimens has been shown to work conveniently and reliably.

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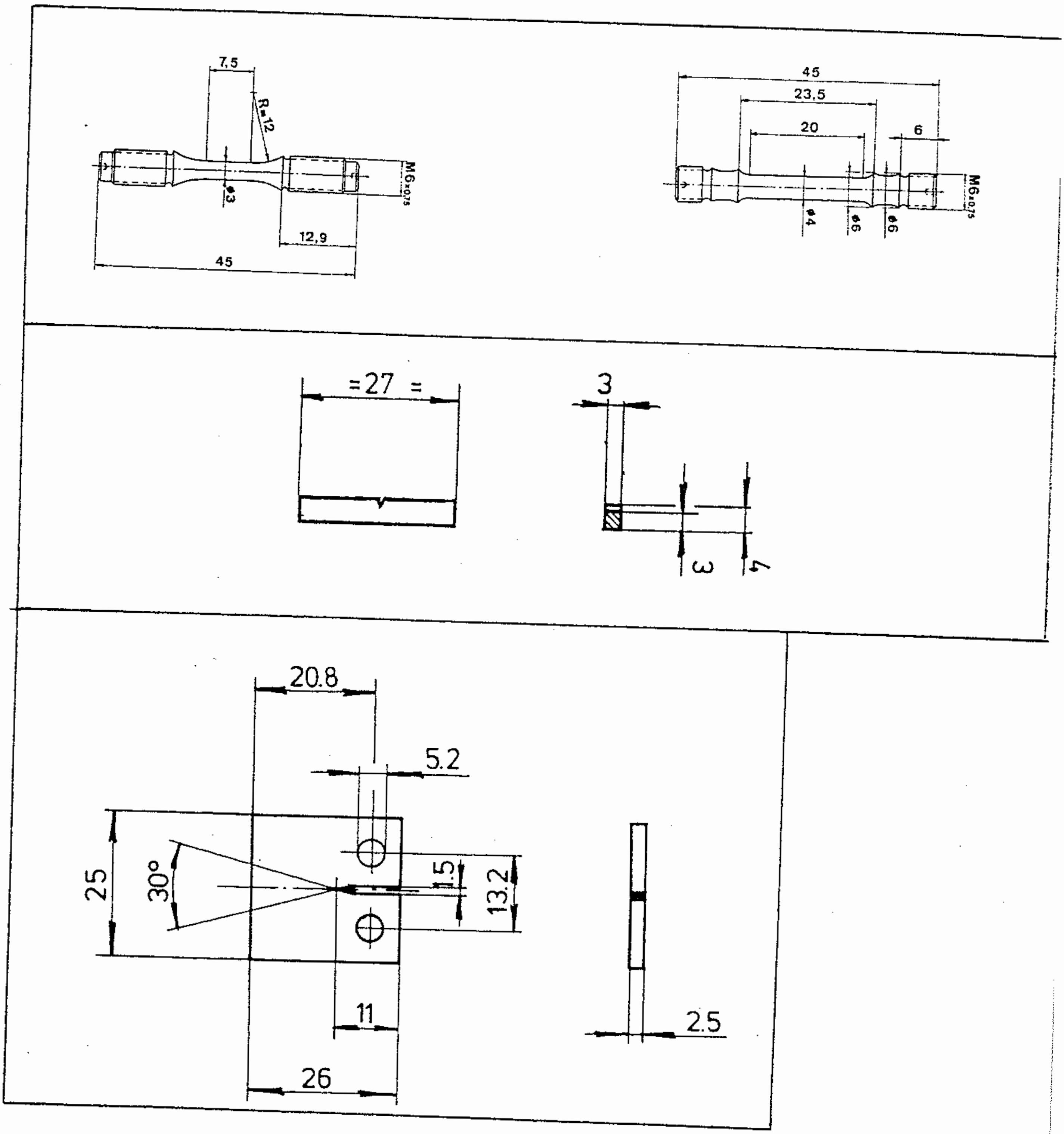

Figure 1 Geometry and dimensions of various types of miniature specimen as used at ECN

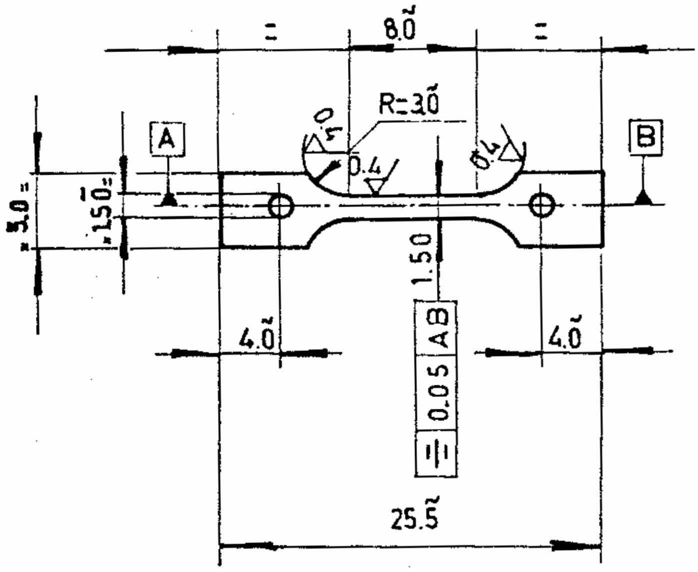

Figure 2 Geometry and dimensions of miniature tensile specimen

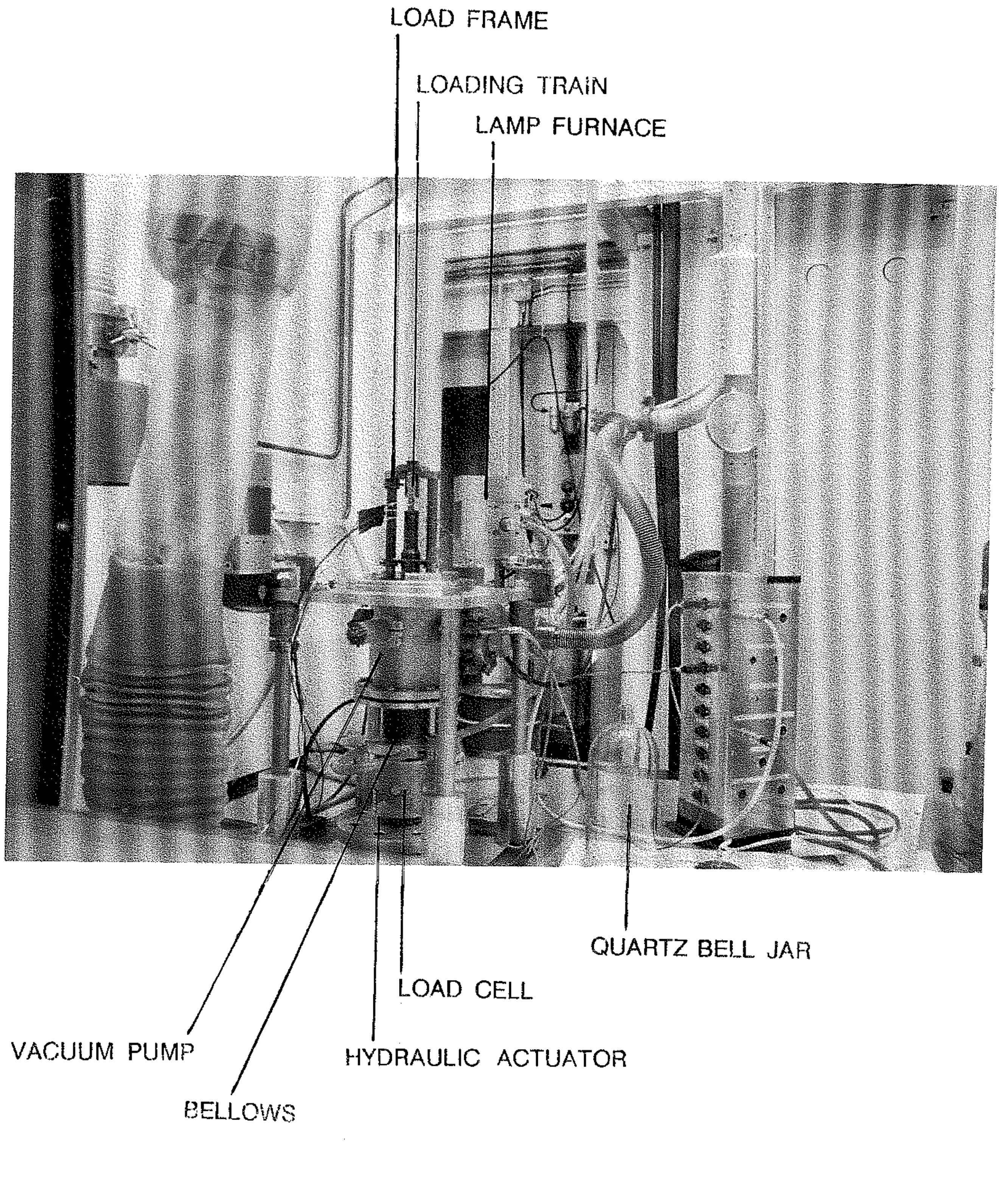

Figure 3 A view of the testing facility

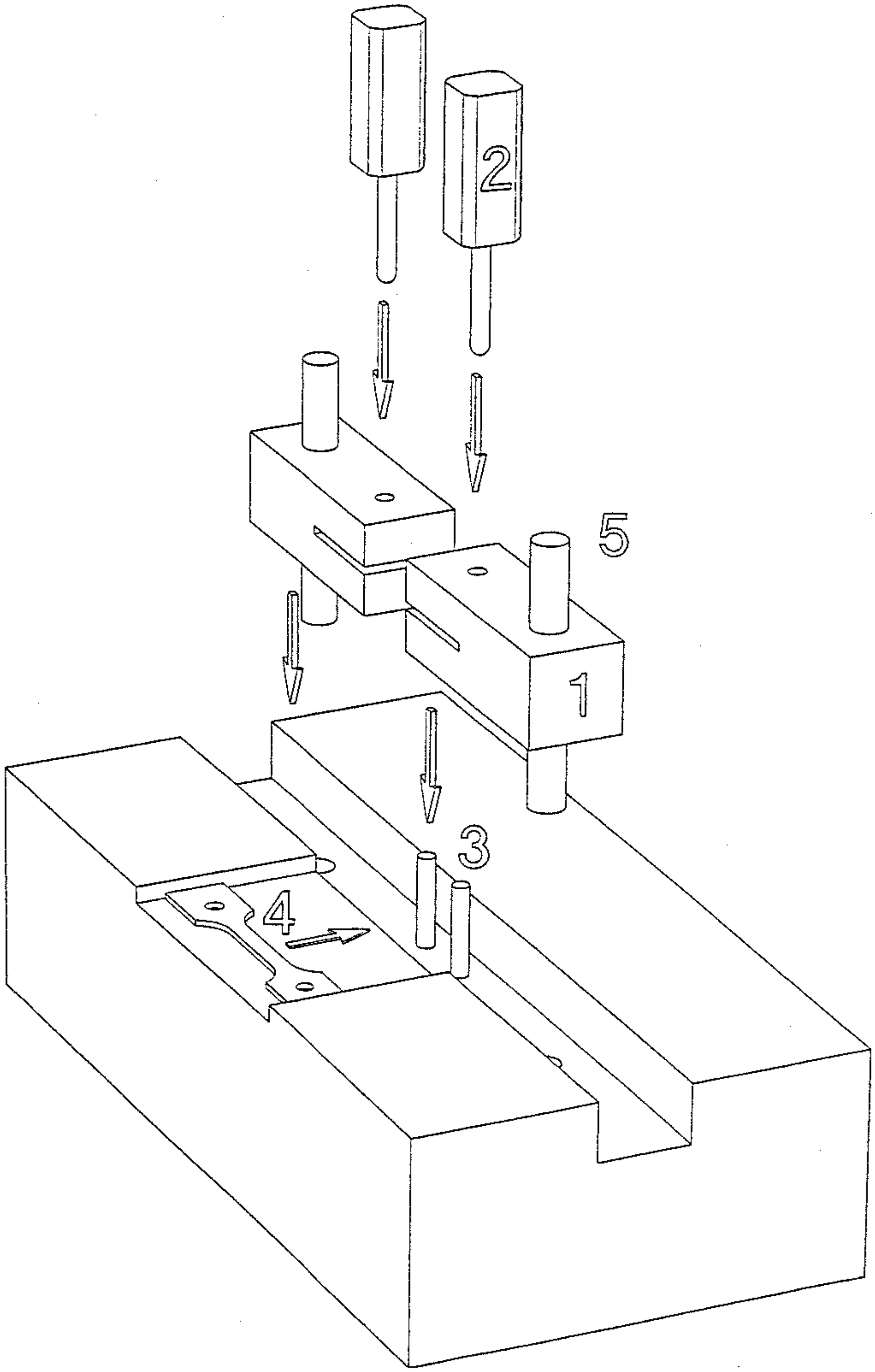

Figure 5 Schematic of assembly template for miniature specimens

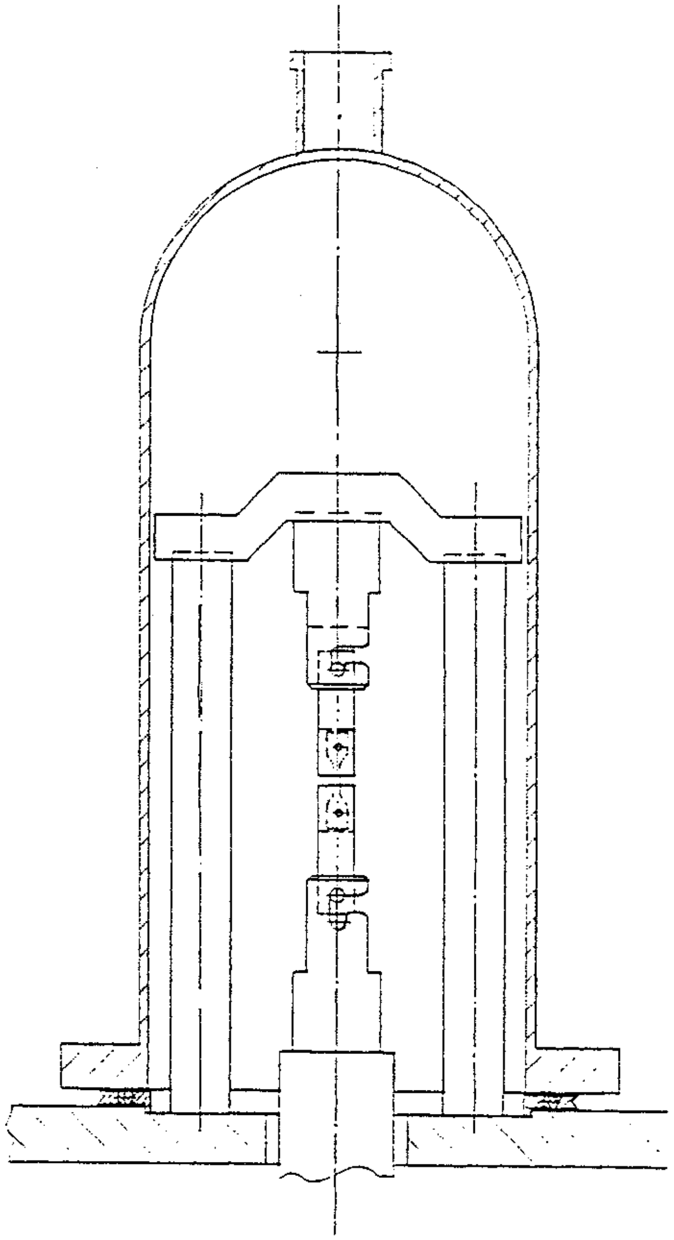

Figure 4 Schematic of testing apparatus