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A MAGNETIC SUSCEPTIBILITY BALANCE SUSPENSION*

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ABSTRACT

A thermocouple has been used as the suspension in a magnetic susceptibility balance. This construction is described and some measurements providing an assessment of the performance of the balance are given.

INTRODUCTION

One of the difficulties associated with the measurement of paramagnetic susceptibilities, especially when these are small, by force methods is the choice of a suitable material of appropriate dimensions as a suspension to suspend the paramagnetic material in the magnetic field. The solutions generally provided to this problem range from fine quartz filaments through quartz tube to metal tube and metallic rod. The quartz filament suspension offers the closest approximation to a free specimen which is almost isolated from its surroundings, so that when it is used the force measured is effectively the force acting on the specimen material alone except when the specimen material is available only in small quantities and has a small susceptibility. The major disadvantage of the quartz filament suspension is that the temperature of the specimen can be measured only by placing a thermometer (generally a thermocouple junction) close to the specimen material. When tube suspensions are used, the specimen is generally enclosed in some form of specimen box, and the temper ture of the box is measured by a thermometer in thermal contact with it. Since the specimen is likely to be in fair thermal contact with the box, it is quite reasonable to expect that the measured temperature of the box is that of the specimen. The tube-specimen box system, however, involves a large quantity of "background material" which gives rise to forces in addition to the force acting on the specimen material: the specimen force might even be small in comparison with the "background" force. This type of suspension also involves the necessity of calibration in the absence of a specimen and the annoyance of having to subtract the "background" force

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(which may vary appreciably with temperature) from every force measured during a set of measurements.

METHOD

The purpose of this paper is to describe a force measuring system, which represents a solution to the suspension problem which is a compromise providing some of the advantages of both tube-box and filament suspensions. The basic idea is simply that the thermocouple, which is made from fine wires, acts as a filamentary suspension. The specimen, in the form of a cylinder with an axial hole, is located at the thermocouple junction and surrounds it, so that the temperature measured by the thermocouple is the temperature inside the specimen. This configuration was used because the capacity of the Cahn RG electrobalance used as a force measuring device was 1 g. The thermocouple was attached to the central pivot of the balance and taken along the weighing arm to the suspension point: it was glued in position there and hung vertically down from that point. In order to avoid the difficulty that it might not hang vertically, the thermocouple wires were twisted together. An aluminium weight pan was glued to the thermocouple near to the suspension point to provide a platform on which calibrating weights could be placed. The thermocouple wires extended well below the thermocouple junction, and the specimen was positioned so that the junction was as its centre; the specimen was supported by a small block of Johnson-Matthey Specpure copper which had two mutually perpendicular holes drilled in it. The thermocouple wires passed through one of these holes and the other carried a cotter pin of Specpure copper which held the copper block firmly on the thermocouple well below the junction.

The thermocouple used was 0.03% Fe–Au/Cu, the wires being of 0.08 mm

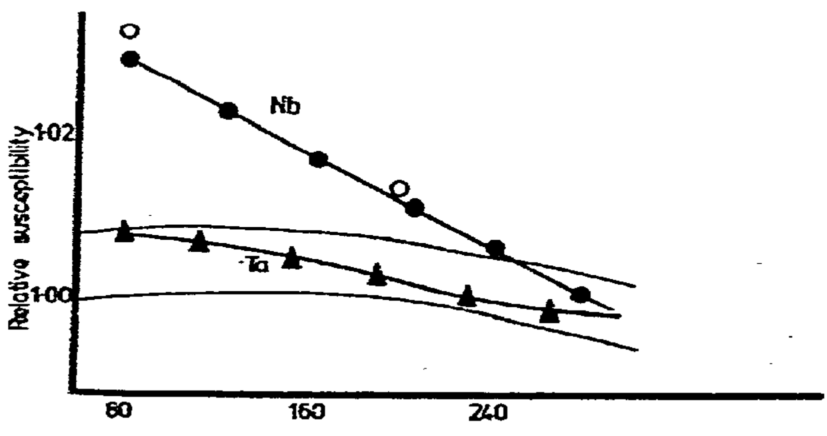

Fig. 1. The relative susceptibilities (χ/χ_{300}) of tantalum and niobium. The lines represent results obtained in this laboratory and the open circles represent points for niobium taken from the work of Asmussen and Soling[2]. The dotted lines represent the standard deviation of 0.4% from the equation of the line for the relative susceptibility of tantalum given by Hoare et al.[1].

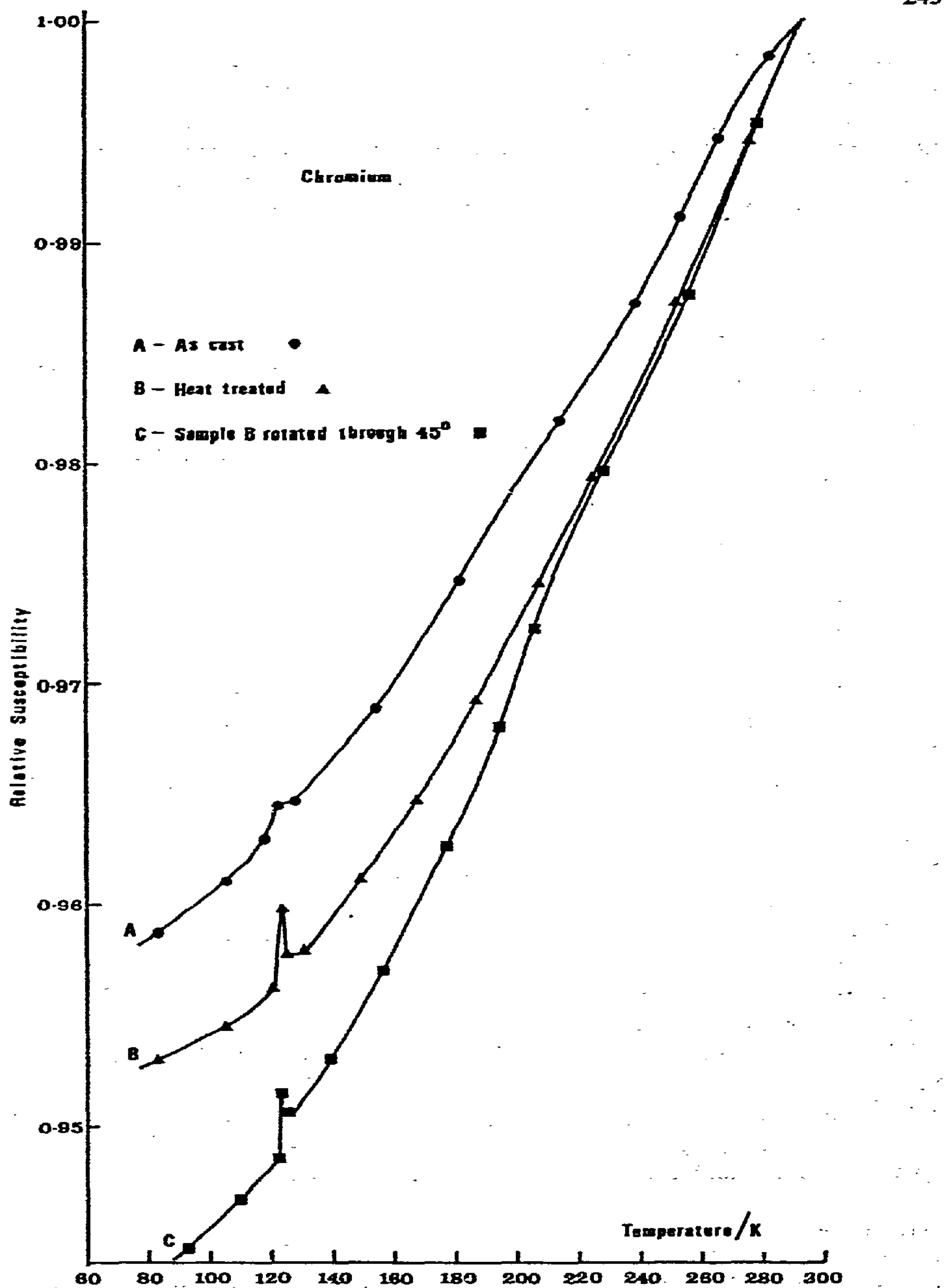

Fig. 2. The relative susceptibilities (χ/χ_{300}) of a specimen of Metals Research chromium. The temperature at which the discontinuity occurred for the annealed specimen was 123.5K.

diameter, and enamelled. The force acting on this thermocouple (and copper block) was found to be small and varied little with temperature, ranging from 30 μg at room temperature to 32 μg at 77K. This force was about $\frac{1}{2}$% of the total force measured and its variation of the order of 0.03%. The thermocouple was calibrated for temperatures down to 77K by immersing the junction in freezing mixtures and liquid nitrogen whilst it was suspended from the balance. The magnetic field used in the measurements was produced by a Newport 7" electromagnet; the current supply to the magnet was stabilized to at least one part in 10^4, providing a constant current of about 10A producing a central field of about 7500 oe.

The performance of this equipment can be judged from the results of measurements made on some standard materials whose susceptibilities have been measured independently in the temperature range 300–77K. Two such materials are tantalum[1] and niobium[2]. Hoare's results for tantalum were presented as a least squares fitted quadratic form of the relative susceptibility (χ/χ_{293}) with a standard deviation of 0.4%—this is compared with measurements made on a specimen of Johnson-Matthey "Specpure" tantalum in this laboratory in Fig. 1, whence it is seen that these results fall within the standard deviation of Hoare's results (shown by the dotted lines). Since the mass susceptibility of tantalum is $0.849 \cdot 10^{-6}$ g^{-1}, the agreement here is to within about $3 \cdot 10^{-9}$ mass susceptibility units at 77K, and generally considerably better. The Asmussen–Soling results are more difficult to interpret since they present a mean square fit to their results, but omit any mention of the standard deviation. The points marked for niobium in Fig. 1 are taken from the Asmussen–Soling line, but when it is recollected that the mass susceptibility of niobium is $2.3 \cdot 10^{-6}$ g^{-1}, it is seen that the deviations between these results (for Specpure niobium) and those of Asmussen and Soling correspond to about 10^{-8} mass susceptibility units at 77K.

The accuracy of temperature measurements can be judged from the results shown in Fig. 2, of measurements which were made on chromium (5N) supplied by Metals Research Ltd. The specimen was machined from a cold hearth melted polycrystalline rod and measured in the "as received" state. It was then annealed in an A.C. furnace, in vacuum, at 1000°C for a week and slow cooled (30°C h^{-1}). This annealing led to marked grain growth and orientation which produced a discontinuity in the susceptibility at the spin flip temperature in further measurements. The spin flip temperature was measured as 123.5K whilst that given by Pepper and Street is 123K. The appearance of curve B which is almost identical with that of Pepper and Street's ⟨110⟩ measurements[3] suggested that a different form of χ–T curve might be obtained by rotating the specimen through 45° about its axis. The resulting χ–T curve (curve C in Fig. 2) can be explained qualitatively in terms of the antiferromagnetic domain structure and grain orientation of the chromium.

REFERENCES

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