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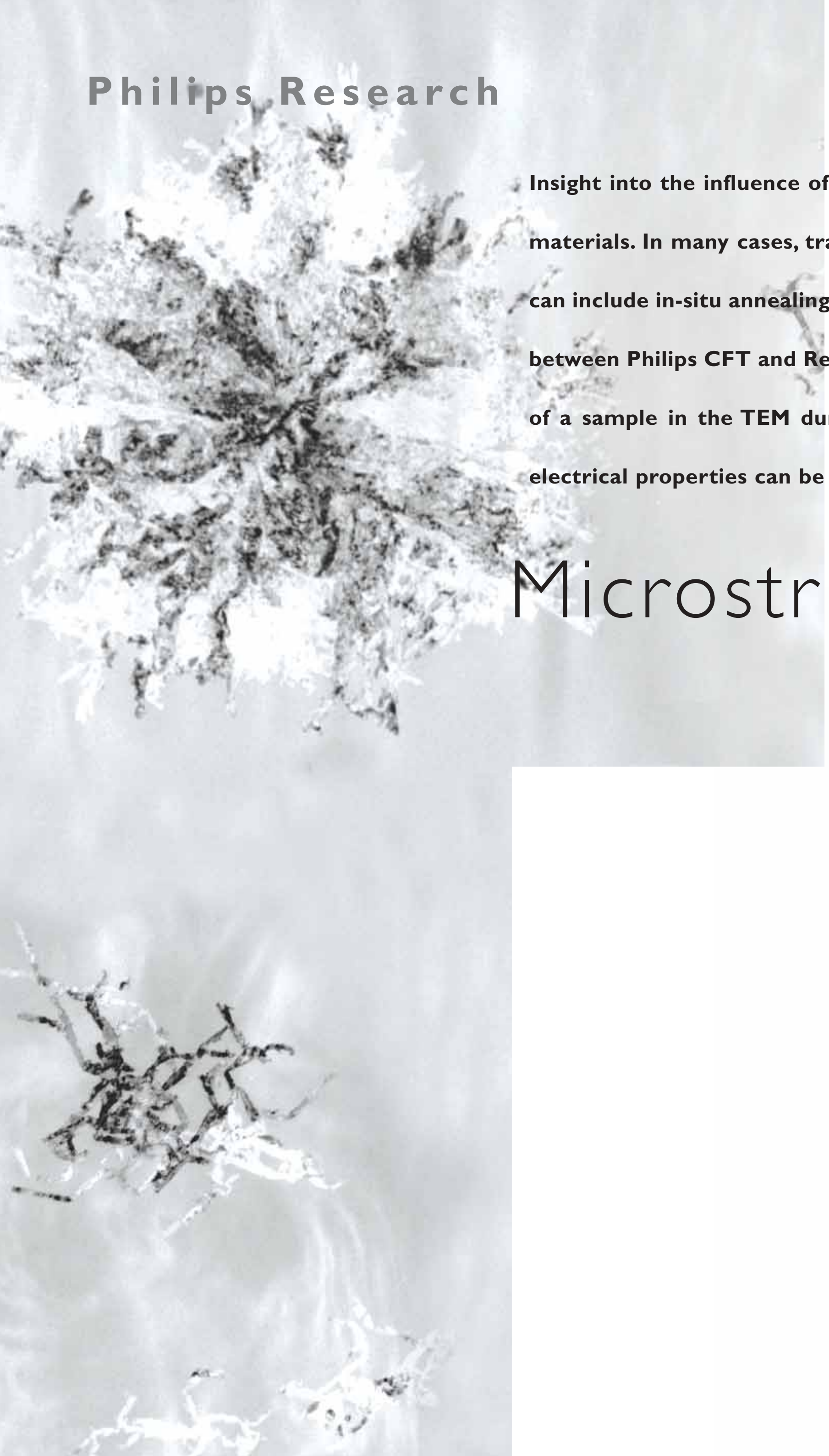

Insight into the influence of the microstructure on the electrical properties is of utmost importance for the development of various materials. In many cases, transmission electron miscroscopy (TEM) is very useful as it has an extremely high resolution. TEM studies can include in-situ annealing experiments of the material of interest to monitor changes as a function of temperature. In collaboration between Philips CFT and Research, a dedicated specimen holder was developed that enables measurement of the electrical resistance of a sample in the TEM during annealing. In this way, a direct correlation between changes in microstructure and changes in the electrical properties can be obtained.

Microstructure vs electrical properties

In-situ resistance measurements as a function of temperature in the TEM

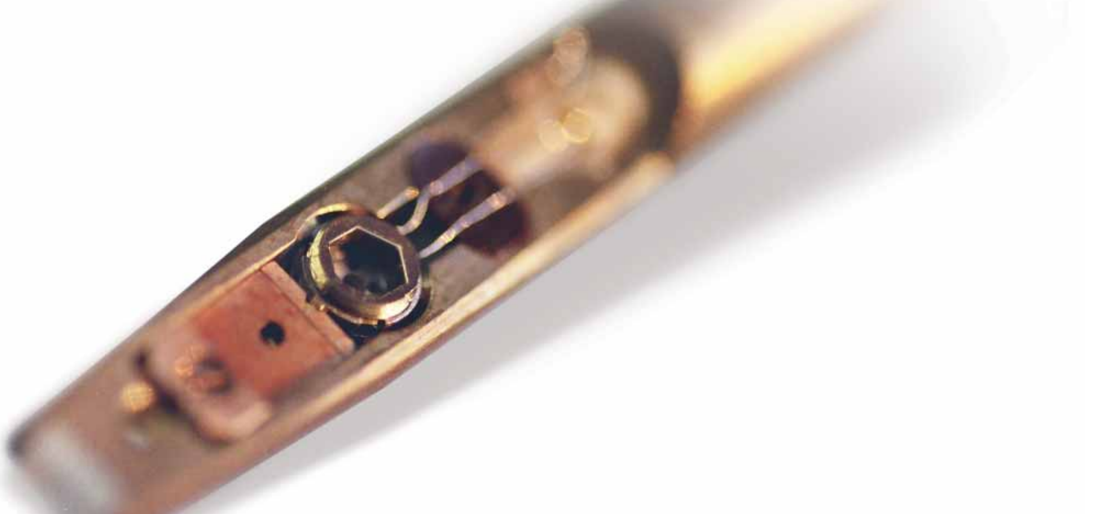

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Fig. 1: TEM specimen holder for resistance versus temperature experiments.

Al-Ge alloys

Sputter deposition of thin films consisting of Al-Ge alloys yields amorphous layers. When the material is heated, crystallization occurs at approximately 470 K, depending on the stoichiometry of the film. During this phase transition the electrical resistance drops by a factor of 10^3 to 10^4.

At the moment, several applications of such films are being realized in the field of micro-electronics. For instance, low ohmic interconnect lines can be made in a high ohmic layer of Al-Ge by heating the material with a laser. This means that parts of the circuitry which were disconnected until then can be activated. In a similar way, a thin film potentiometer has been realized.

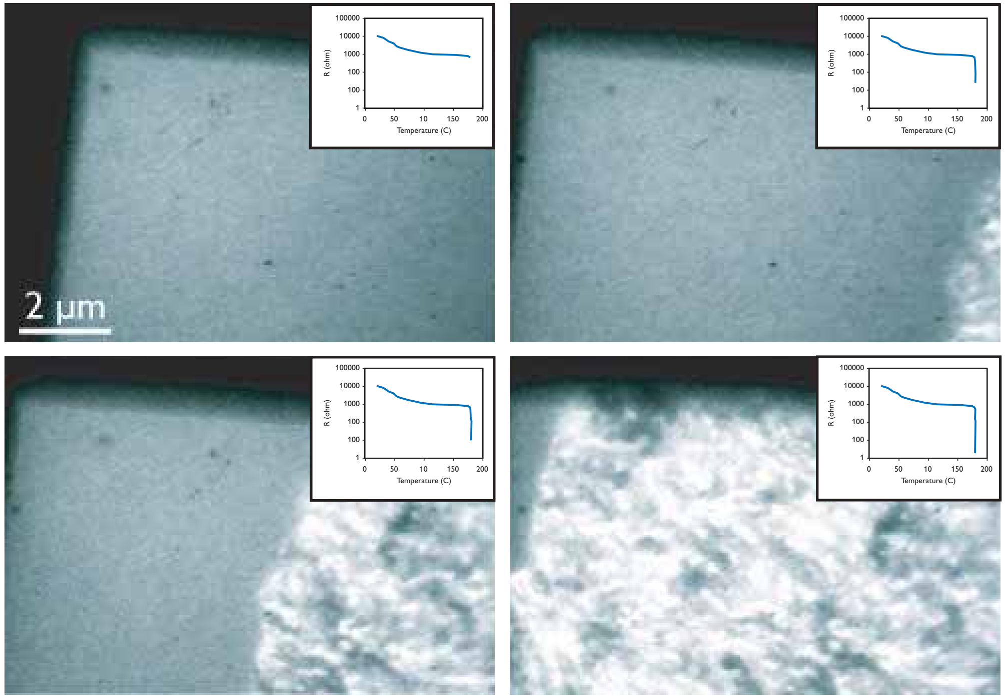

Fig. 2: Video images simultaneously displaying the crystallization of an Al-Ge film and the accompanying drop in electrical resistance. In the insets the R-T dependence is shown on a logarithmic scale.

Phase transition studies

For TEM studies thin films of a 50:50 Al-Ge alloy were sputter-deposited on amorphous Si_3N_4 membranes. Using the special specimen holder (see figure on front page and figure 1), heating experiments were performed. During these studies the microstructure and electrical resistance were simultaneously recorded on videotape. The result is shown in fig. 2. In the first snapshot an amorphous film with a high resistance is visible. The next three snapshots show both a decrease in resistance and a change in microstructure. A crystal growth front runs laterally through the film from right to left. The crystalline part shows a large spread in contrast. This is due to the fact that this part is polycrystalline and all crystals diffract the electron beam differently.

Where fig. 2 shows a detail of the growth front, fig. 3 shows an overview of the TEM sample. From this micrograph it is clear that during the phase transition many crystalline nuclei are formed, all growing out radially. When a chain of these (well-conducting) crystalline regions is formed the resistance shows a sudden drop of several orders of magnitude.

The fact that these crystalline regions are formed simultaneously all over the sample is indicative of the homogeneity of the temperature over this sample. As a result, a direct correlation between crystallization of the Al-Ge film and its electrical behaviour was demonstrated using the dedicated specimen holder.

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For more information:

Tel./fax: +31 -40-27 43210/43075
E-mail: materialsanalysis@philips.com
http://www.philips.com/materialsanalysis
http://pww.natlab.research.philips.com:25222/

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Marcel Verheijen

Fig. 3: During crystallization several nuclei appear in the film, each nucleus growing out radially. The nuclei appear at random sites.