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Defect analysis with atomic-resolution microscopy

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Summary

This work concentrates on the manner in which defects are manifest in a number of materials. Often, the influence defects have is determined by measuring 'average properties'. The techniques utilized are both sensitive and coarse. The actual presence of defects is then assessed by models. In the research described in this thesis, however, the atomic defects are examined by direct methods. This is performed by utilizing microscopes with sufficient resolving power to identify single atoms: Field-Ion Microscopy (FIM) and Scanning Tunnelling Microscopy (STM). These instruments are not only sensitive, but also fine-scaled. Only a small amount of material actually contributes to the image formation. This approach has given rise to new insights.

Field-ion microscopic investigations of twins in YBa₂Cu₃O₇₋₈

In the high temperature superconductor $YBa_2Cu_3O_{7-\delta}$, being examined by field-ion microscopy, it is found that a particular planar defect, the twin boundary, has a much larger influence on the acquired images than might be expected on the basis of model calculations. The calculations are performed with the assumption that the twin boundary separates two parts of the material being each other's mirror image. The field-ion micrographs strongly suggest a disordered boundary region. This may be explained from the fact that the oxygen atoms, being aligned in chains in the perfect structure, are disoriented in the vicinity of the boundary, where they not only are subject to the influence of neighbouring chains, but also of perpendicular chains in the opposite region.

Field-ion microscopic investigations of the structure of decagonal quasicrystals

Results of diffraction-experiments on decagonal quasicrystals strongly point in the direction of a framework, originally devised by Penrose. In order to obtain information on the precise atomic configuration within such a framework, the field-ion microscope is utilized. With this instrument, imaging of decagonal quasicrystals, obtained through rapidly solidification from the melt, was accomplished. To facilitate interpretation of field-ion micrographs, a framework model of stacked Penrose-tilings was used to calculate field-ion micrographs. The actual field-ion micrographs showed hardly any resemblance with the calculated counterparts. The reason for this may partially be sought in the invariable presence of aluminium in decagonal quasicrystals. The weak binding of alu-

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minium onto the crystal surface hampers stable imaging. Determination of the exact atomic configuration therefore seems to be a too difficult task for the field-ion microscope. A more principal impediment lies in the assessment that the periodic axis is invariably perpendicular to the emitter axis and an image corresponding to the Penrose-tiling may not be obtained.

Various regimes of charge density waves in transition-metal dichalcogenides

A number of transition-metal dichalcogenides (TMDC) are found to exhibit stationary lattice distortions. Along with these periodic lattice distortions (PLD), the electron density is spatially modulated (Charge Density Waves, CDW). These PLD/CDW phenomena are comparable to the Peierls distortion encountered in one-dimensional metals. With the STM it is possible to determine, under certain circumstances, the CDW part of the distortion. In the case of TiS_2 , that exhibits no global CDW, it is seen that the 'normal' crystal surface is imaged along with some marked triangular features and deep-lying holes at specific sites. It is concluded that these features correspond to defects present in the material. By determining the exact sites at which the features occur and the relative occurrence a model is proposed in which a number of titanium atoms is displaced. Displaced titanium atoms already appear in earlier defect studies , the sites to which the atoms are displaced, however, differ from earlier conceptions.

When the exact shape of the features is observed, a decaying wave-like nature is encountered. The question to be solved is whether these features are connected to the CDW phenomenon. By using expressions derived from a Ginzburg-Landau formulation for the charge density around impurities, earlier devised by McMillan, a striking outcome is encountered: For all three features observed by STM there exists a set of parameters for which the calculated charge density resembles the observed one significantly. The chemical nature of the defect is not included in the expressions for the CDW and therefore it is not proved that displaced titanium atoms are responsible for the observed features. The agreement between calculation and observation, however, urges considerable notion to be given to this explanation. In addition, another TMDC (NbSe₂) is responsible for images that may be considered to lie in between the global CDW and the localized CDW in TiS₂. It is probable that the formalism used in the localized charge density wave may be applicable here as well. The demonstration of this is, however, impeded by the vast number of possible sources.

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Design, implementation and performance of a combined STM/SEM instrument

With STM we are able to observe, in atomic detail, solid surfaces, which has been utilized in the foregoing. When the practical aspects of STM use are considered it is conjectured that the attainable resolution is considerable, but that the field of view is limited to about one µm. This impedes certain research tasks, such as in which a characteristic, though rarely occurring feature like a grain boundary is to be examined. To arrive at a solution to this problem, a combination between STM and another existing microscope, the SEM is evaluated. The resolving power of the SEM is not sufficient to resolve individual atoms. The field of view, however, is large enough to use the SEM as a guide to the STM-probing tip. Alternatively, the STM may be operated as a high-resolution stage to the SEM. Proceeding along this thought, a design was made based on an existing SEM and a newly developed STM. The STM has to comply with a number of specific requirements. These requirements are converted into a design which was subsequently implemented. A transition-metal dichalcogenide and sputtered (rough) gold surfaces were taken as testspecimens. It is shown that the STM is capable of attaining atomic resolution. Furthermore, the location of the STM-probe may be monitored by means of SEM. Simultaneous tunneling with the STM-probe and SEM operation has been shown feasible. In conclusions it may be assessed that the combined instrument has accomplished a successful synthesis between the two complementing techniques.

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