



Research Article

Towards YMD (Y-Modulation Detection Algorithms and Devices) Applications in CryoSEM for Surface Studies of Superconductors: Per Aspera ad Astra

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Abstract

The use of various electron microscopy techniques to study superconductors has been established since the 1980s, encompassing both high-resolution transmission electron microscopy (HRTEM) and lower magnification scanning electron microscopy (SEM). A significant challenge with these methods is the temperature dependence of imaging within the electron microscope, which necessitates noise reduction and specialized cooling systems, such as Low-Temperature Scanning Electron Microscopy (LTSEM). Programmable stages with temperature stabilization allow for analyzing temperature dependencies similar to studies on dielectric composites and semiconductors. Attempts to replace SEM and HRTEM with simpler scanning probe and tunnelling microscopy methods have not resolved the initial challenges, as they still require advanced cooling setups. The SEM benefits of scanning for large area investigations with satisfactory temporal dynamics are often overlooked. The interaction of the superconductor with the electron beam not only visualizes but can also contrast and modify samples, revealing cooperative or additive defects observable at micro- and mesoscopic levels. Therefore, it is advisable to study the charge structure of the surface of high-temperature superconductors at real temperatures. The proposed methodology involves using Y-modulated detection techniques with JEOL microscopes, applicable in both Charge Collection Scanning Electron Microscopy (CCSEM) and electronic spectroscopy methods. This approach allows for detailed 3D topographical imaging of surface charging structures/textures created by charged structures.

Introduction

The use of various electron microscopy techniques for studying superconductors has been common since the 1980s, both for high-resolution transmission electron microscopy [1,2] and for scanning electron microscopy with lower magnification and resolution [3,4]. A significant problem for such methods is dependence of the image quality on the temperature and beam parameters, which requires noise suppression and specific cooling (LTSEM – Low-Temperature Scanning Electron Microscopy) [5–7]. When using a programmable table with temperature stabilization [7], it is possible to analyze temperature dependences in the same way as in the case of studying dielectric composites and semiconductors [8]. The attempt to replace SEM and HRTEM with simpler methods of scanning probe [9,10] and tunnel [11,12] microscopy, from our

point of view, did not lead to a conceptual progress, since it is still necessary to modify and use cooled tables and cryogenic modules, in particular, those that differ from the standard modules produced for cryopreservation in cryobiology, for cryochemical or cryophysical tasks [13–16].

The tasks of establishing dependencies of the microscopic structure and electronic defects [17] are solved in tunnel, probe, and atomic force microscopy extensively due to the acceleration in the kinematics of the probe/cantilever. At the same time, the advantages of scanning as a method for studying large sample areas with satisfactory time dynamics (as it has been since the advent of the TV mode in SEM) are not used in such cases. Nevertheless, it is obvious that the electron beam can not only visualize, but also contrast and modify the sample, which, from the standpoint of solid state physics, corresponds

to a number of the possible defects of a cooperative or additive nature manifested not only at the subnanometer level, but also at the SEM-resolved micro- and mesoscopic level.

For this reason, it is advisable to study the charge structure of the superconductor surface with the subsequent analyzing of the scangrams in a mode resolving the surface texture using the selection of several isopotential lines performed by the special algorithms.

Methods

We propose to use the YMD technique and equipment ("Y-modulation mode" based on "Y-modulation device") developed for JEOL and Stereoscan (Cambridge Instrument Company) scanning electron microscopes. Y-modulated detection mode is used both in CCSEM (Charge Collection Scanning Electron Microscopy) techniques [18] and in electron spectroscopy methods based on the analysis of the energy distribution of electrons emitted during a nonradiative transition providing relaxation of the excited state, which emerged as a result of the vacancy formation in one of the inner electron shells (Auger microscopy methods [19]).

This mode can also be used in low-temperature SEM techniques [20] (including the analysis of superconductors [21]) and in conventional methods of reconstructing the topographic surface texture [22], according to the same principles as in confocal laser scanning microscopy [23]. The initial image is obtained during YMD scanning (the image sequence in Figure 1), as a result of which it is possible to observe a complete relief/bas-relief picture of the surface texture, created by the height gradient of the charged structures (for example, using the YMD technique, it is possible to distinguish between sources and drains in semiconductor devices by densitometry).

Materials

We have studied the samples of superconductor ceramics, which are not subject to disclosure as "know-how" in this methodological article. Due to this, in the test experiments, we did not cool the samples up to cosmic cryovacuum temperatures. However, as a motivation for the analysis and visualization of superconductors at lower temperatures (literally simulating cosmic cryovacuum conditions), we can refer to the works on the detection of superconductors and superconductivity phenomena in micrometeorites, as well as in the fragments of large meteorites that fell to Earth [24–27].

Results

Several negative and positive image series of the field disturbances and charge wandering in the form of travelling waves and solitary waves in a potential superconductor, registered by SEM with a Y-modulating device without cooling, are shown in Figures 2 and 3. It can be seen that not only single waves are observed, but also fine modulations of the charge distribution surface profile, varying significantly in time. There are both stationary and regular or repeating components of the wave behaviour (depending on the surface structure or texture), and modulated spontaneous bursts and wave

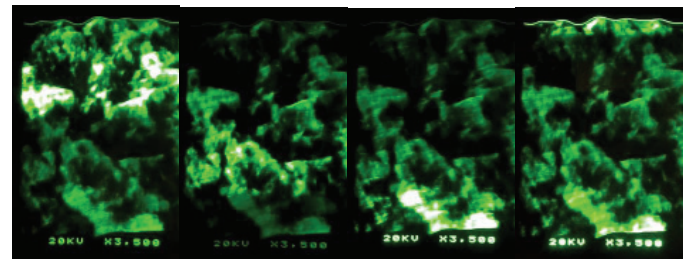


Figure 1: The scanning process of a superconductor surface in the Y-modulation detection mode before any processing and decoding. One can see the change in the positions of the luminance maxima ("3D Luminance Surface"), corresponding to the line scanning.

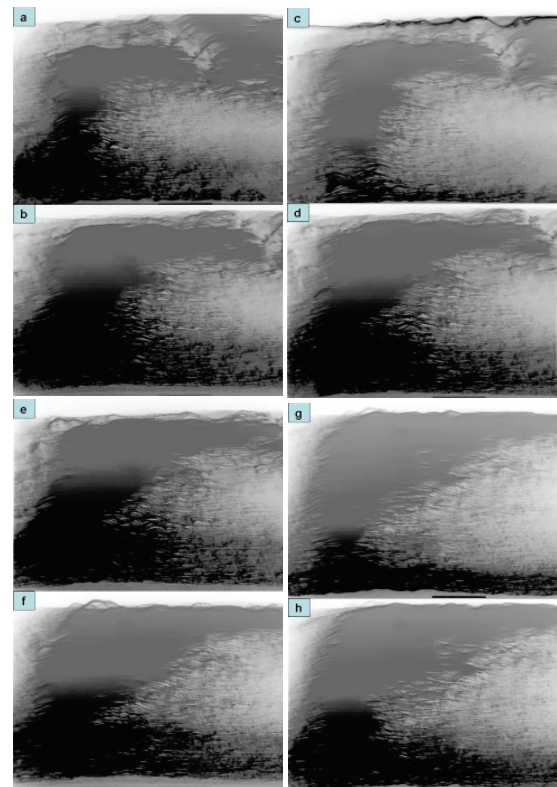


Figure 2: Positive images of YMD visualizations for the possible superconductor material.

fronts. This pool of wave phenomena for an uncooled potential superconductor material (before the phase transition) can be characterized by cumulative dynamics, i.e. an increase in their amplitude, a change in the frequency of bursts and dissipation phenomena until the transition to the superconducting state, in which, according to the definition of superconductivity (the property of some materials to conduct electric current without dissipation with the simultaneous expulsion of a magnetic field), dissipation is not observed. In the verification experiments, the equivalent circuit of a sample with a table or a foil substrate behaves incompatibly with the superconducting state, which confirms the correctness of our approach.

Discussion

The application of this 3D visualization method is possible in several aspects, including modeling of non-classical effects in photosynthesis and tunnelling reactions (including

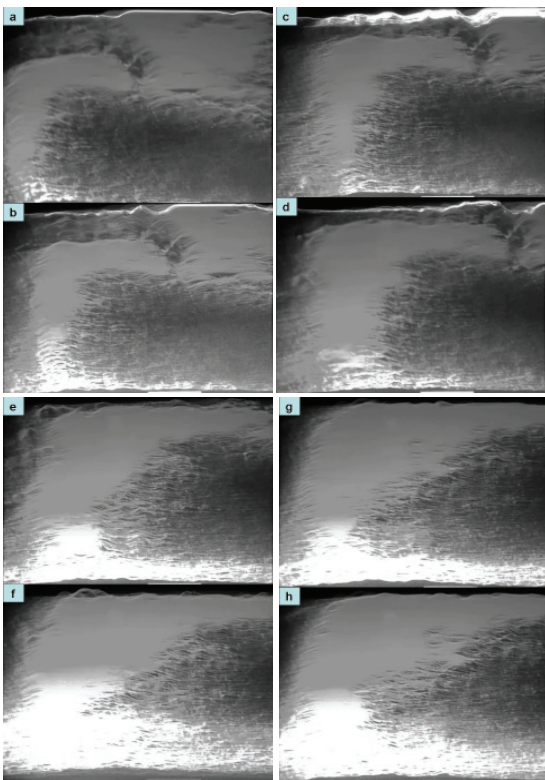


Figure 3: Negative images of YMD visualizations for the possible superconductor material.

prebiological synthesis in cryogenic media proposed by V.I. Goldansky) in mineral samples [28–30], even in space conditions [31–35]. Accordingly, this method can also be applied in cryoelectron microscopy for validating Goldansky's hypotheses and experimental verification of the possibility of templating processes on cosmic bodies, including templating processes on superconducting minerals [36,37]. Therefore, if the development of the proposed YMD visualization technology allows to analyze in real time (in a dynamic or time-lapse / time-resolved mode) the electrophysical state of the natural superconductor surface, this will be a significant contribution to the development and testing of new devices based on superconducting elements using traveling waves – such as, for example, traveling-wave amplifiers [38–42], traveling-wave photodetectors [43–47], traveling wave cavity prototypes [48,49], traveling-wave antennas [50–52], traveling-wave-type optical modulators or optomechanical traveling wave phonon–photon translators [53,54], etc. However, for such a primary communication as this propositional work, it does not seem rational to talk much about the future applications of the proposed technique.

Supplement: What is YMD-EM?

The development of Y-modulation methods in electron microscopy began with the introduction of the first Y-modulated scanning systems (Y-modulation detection and scan-modulation displays) in SEMs in the late 1960s [55–57]. In the pioneering work [55], the authors state:

“In scanning electron microscopy (reviewed by Oatley, Nixon, and Pease, 1965), the signal from the specimen is generally used

to modulate the brightness of a cathode ray tube (c.r.t) to give a television-type display. This display has the advantage of giving a picture similar in appearance to one that would be obtained with an optical microscope, and its information is therefore much easier to interpret. However, the contrast observed relies on the differences in brightness levels seen on the face of a cathode ray tube, and the number of distinguishable levels is limited. Moreover, the relationship between these distinguishable levels or “shades of grey” is qualitative. It is sometimes desirable to be able to study the information in a scanning electron micrograph in a quantitative manner, and for this purpose, a special display technique has been developed. The microscope signal is displayed by deflecting the scanning c.r.t beam a distance proportional to the amplitude of the signal, and hence it is known as scan-modulation display. Other descriptive terms that have been used are Deflection, Frame or y-Modulation”.

An example of an electronic block diagram for a Y-modulation imaging device or scan-modulation display (SMD) from the paper [55] is shown in Figure 4.

An explanation of “how Y-modulation operates at the hardware level, specifically at the electron beam control level” for the “Stereoscan” SEM system is provided in Figure 5 from the paper [56] (“Y-modulation: an improved method of revealing surface detail using the scanning electron microscope”) from the “Science” journal.

According to the above-cited paper [56]:

“As in normal scanning electron microscopy, a finely focused beam of electrons is deflected across the surface under investigation. The number of electrons back-scattered and emitted is a function of the topography and composition of the specimen...; such electrons are accelerated by a wire grid biased to + 12 kV and allowed to strike the surface of a phosphor screen. The scintillation produced is viewed and amplified by a photomultiplier, and the output of the photomultiplier is applied to a cathode-ray tube scanning in synchronization. The scan size of the cathode-ray tube remains fixed at 10.5 cm², but the area of scan on the specimen may be successively decreased, thus

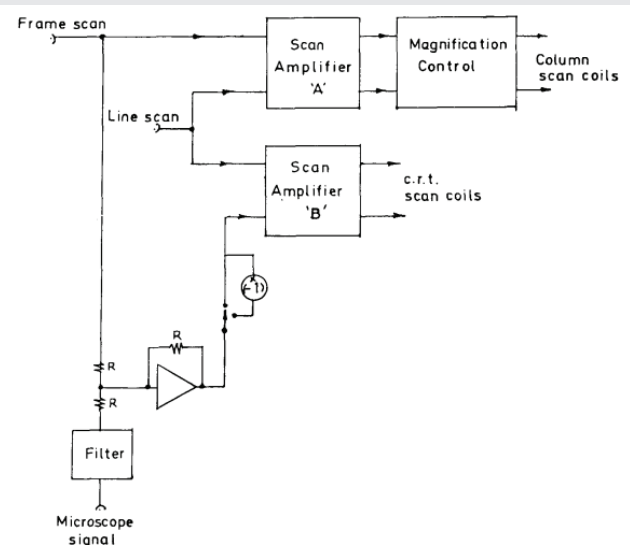


Figure 4: Scheme of scan-modulation display (SMD) from the paper [55].

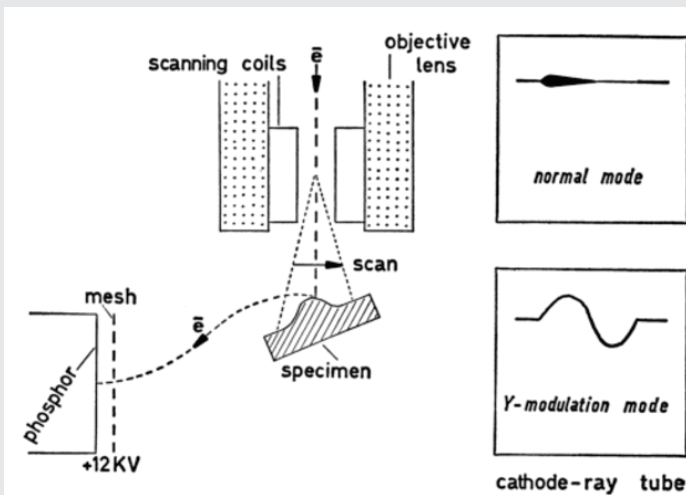


Figure 5: Comparison between standard SEM scanning mode and Y-modulation mode from the paper [56].

giving an apparent step-up of magnification within the range of $\times 20$ to $\times 100,000$ at the normal working distance of 11 mm. These values may be varied either by changing the working distance or by utilizing the zoom magnification factor on the scan rotation unit. "The clarity with which the step-by-step growth features can be observed ... is further exemplified at a magnification which, in the normal mode ..., is clearly unusable. Taken at lower power ..., the Y-shift effect can be seen to introduce considerable difficulties in interpretation if complex morphology or topographical relief is present. Using a lower amount of modulation will reduce this effect..., but, taken in conjunction with the normal mode photograph, the Y-modulation mode photograph permits detailed examination of the fine structure of selected areas. We thus deduce that this method becomes increasingly important as higher and higher magnifications are employed, especially with magnetic materials or on surfaces with low or moderate relief, and that at lower magnification the Y-modulation photographs provide valuable information supplementing normal mode photographs".

Subsequently, Y-modulation techniques were standardized for various detector types, including backscattered electron detectors (BEI) and transmitted electrons, and later for cathodoluminescence as well as for mapping elemental distributions via energy-dispersive X-ray spectroscopy and microprobe analysis [58–60]. This enabled cross-correlation and colocalization of 3D profiles not only for sample topography but also for spatial distributions of their chemical components. Furthermore, quantitative Y-modulated detection and colocalization/correlation of profiles became possible for both STEM (scanning transmission electron microscopy) and conventional TEM within a single instrument without sample removal [61,62]. By this time (the 1985 – 1990s), fully digital signal processing from various detectors and digital implementation of the YMD mode were already available [63], and state-of-the-art electron microscopes were equipped with microcomputer-based imaging systems (e.g., in "new" JEOL JSM models).

Initially (from the 1960s), YMD was implemented using analogue instrumentation—various oscilloscopes with camera attachments and delay lines, followed by storage oscilloscopes. In the 1970s, although equipment from the late 1960s

remained functional, there was a shift toward computer-assisted 3D visualization techniques and computer-mediated YMD. The first developers and users of these technologies were "microelectronics specialists," i.e., experts working with photomasks and photoresists, who required regular, reproducible, and automated measurements of photoresist thickness, photomask features, and etched grooves [64,65]. By the 1990s, YMD was widely applied in both digital and analogue microelectronics (for quality control of integrated circuits), as well as in semiconductor physics and engineering (e.g., for profiling heterojunctions in field-effect transistors) [66,67].

Following the implementation of digital methods that enabled the colocalization of data from any type of scanning on a single image and the construction of 3D profiles / "bas-relief" representations / visualizations of arbitrary data sources related to surface texture and micro-roughness, it became possible to use YMD in synchronized systems of atomic force microscopy and scanning electron microscopy, as well as in tunneling microscopy and scanning electron microscopy; a similar statement holds for synchronized and correlative systems of scanning tunneling (or atomic force) and reflection electron microscopy [68]. The latter assertion seems trivial, since, by definition, Y-modulation scanning in scanning electron microscopy pertains to deflection and reflection scan modes.

In the semiconductor industry and semiconductor physics, YMD visualization is actively employed for visualizing the spatial distribution of defects in semiconductors using a scanning probe and electron beam to excite defects [69] (SDTLS = Scanning Deep-Level Transient Spectroscopy); however, in this case, additional measurements of transient capacitance and current after excitation are used, depending on temperature, which yields quantitative data on the energy levels, concentration, and other characteristics of defects (such as carrier capture cross-sections or activation barriers) in the semiconductor. Papers on YMD visualization in scanning electron microscopy of charging effects on semiconductors are well known since 1970 [70].

To our knowledge, before our unfulfilled proposal (which formed the basis of this article), there were no studies utilizing YMD for cryo-SEM of superconductors.

Author contributions

Aleksandrovsk P.L. – Automation of the JEOL JSM scanning electron microscope with YMD; 3D fabrication of an adapter for a digital camera. Designing and soldering electronic circuits and connecting cables (2018–2019, 2025–2026) [71–74].

Filippov M.K. – Restorations of the vacuum systems of the JEOL JSM scanning electron microscope with YMD mode, cleaning of the scanning electron microscope column, design of a new power supply source, replacement of technical oils (2018–2025). Memoirs about old SEM instruments with Y modulation devices (1970–1990).

Gradov O.V. – YMD experiment design, Cryo-SEM instrumentation development and modernization



(unfortunately, destroyed now) [75–77]. Conceptualization and writing of the review text | introduction part. Time-resolved SEM and time-resolved SEM-YMD experiments on JEOL JSM-based setups (for example, see [78–82], etc.).

Maklakova I.A. – Time-resolved SEM and time-resolved SEM-YMD experiments on JEOL JSM-based setups (for example, see [78–82] etc.). Primary image processing (monochrome mode; contrast; brightness; cropping; etc.).

Conflict of interests: The authors declare that they have no conflict of interest.

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We sincerely thank the late Professor Trusevich for the opportunity to attempt SEM studies of the superconductors (he created). We are deeply concerned about not knowing the composition and are unable to interpret the data or complete the study of charge effects using machine learning methods. Therefore, we are forced to publish only preliminary results demonstrating that our YMD method is, in principle, viable. Unfortunately, due to strict sanctions, we are currently unable to complete the setup and finalize the instrumentation and software developments. Due to the sudden death of the aforementioned co-author, we have not yet developed cryo-electron microscopy techniques with machine learning for these materials.

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