Scripta Materialia 246 (2024) 116009

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.journals.elsevier.com/scripta-materialia

Viewpoint article

Driving next generation manufacturing through advanced metals characterisation capability

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ARTICLE INFO

Keywords:
Microstructure characterisation
Digital manufacturing
Optical microscopy
Electrochemical etching
Acoustic spectroscopy

ABSTRACT

Understanding the effects of manufacturing methods upon materials has driven constant innovation for over 300 years. While our ability to fabricate metallurgical wonders extends into the annals of history our ability to understand the scientific principles where process meets material has been pivotal to improving our capabilities. In this letter we briefly consider this history, comment upon the current state-of-the-art and, most importantly, propose new technologies for future industrial application which have been devised and exploited by the authors. It is hoped that this letter will allow other researchers to engage in this topic and facilitate the emergence of new processcompatible technologies which do not require destructive evaluation. This is particularly timely given the ability to manipulate microstructures with increasing dexterity. This is perhaps best illustrated in additive manufacturing [1] but is also a key consideration when process planning for machining [2], grinding [3] and forming [4].

Understanding the effects of manufacturing methods upon materials has driven constant innovation for over 300 years. While our ability to fabricate metallurgical wonders extends into the annals of history our ability to understand the scientific principles where process meets material has been pivotal to improving our capabilities. In this letter we briefly consider this history, comment upon the current state-of-the-art and, most importantly, propose new technologies for future industrial application which have been devised and exploited by the authors. It is hoped that this letter will allow other researchers to engage in this topic and facilitate the emergence of new process-compatible technologies which do not require destructive evaluation. This is particularly timely given the ability to manipulate microstructures with increasing dexterity. This is perhaps best illustrated in additive manufacturing [1] but is also a key consideration when process planning for machining [2], grinding [3] and forming [4].

A historical overview - Advances in materials science of the late 19th and early 20th centuries were driven by our new understanding of the atom, its role in defining crystal and therefore material properties, and, of importance to this perspective, the physics of early materials characterisation tools [5]. At this time, Henri Becquerel alongside Marie and Pierre Curie were providing new insight to the nature of radioactivity by observation of 'Becquerel rays' later to be known as radiation [6]. Shortly thereafter, at the Cavendish lab in the United Kingdom, Ernst Rutherford reported peculiar phenomena resulting from the transmission of radioactive particles through thin metallic sheets [7]. This would be explored as X-Rays (or Röntgen) by Max von Laue and later related to crystallographic parameters by father and son W.H and W.L Bragg [8] giving rise to our modern day understanding of diffraction-based characterisation methods.

Similarly, Thompson's discovery of the negative charges associated with the atom (electrons) and the means by which these cathode rays could be manipulated in a magnetic and electrical field [9] provided the basis for electron microscopy.

It was the combination of diffraction and electron microscopy which gave rise to modern materials characterization practices. These enable scientists to connect the atomic and micro-structure of materials to their properties.

In the pursuit of understanding the structure of matter, the breadth of wavelengths we use for characterisation tools has increased dramatically. This was initiated in rather humble beginnings. Presumably, since Newton's first microscopic observations [10] and early metallurgists following in Sorby's footsteps turned their microscopes to metals to

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https://doi.org/10.1016/j.scriptamat.2024.116009
Received 15 November 2023; Received in revised form 21 January 2024; Accepted 25 January 2024
Available online 3 February 2024

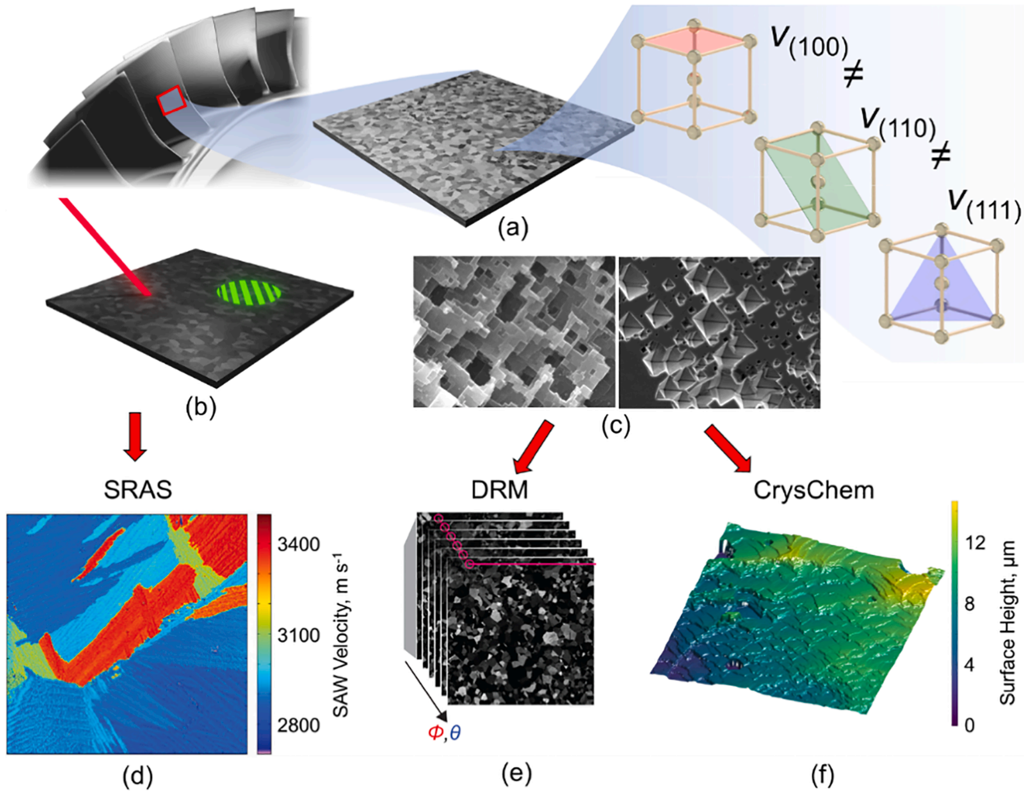

Fig. 1. Exemplar technologies which provide microstructural information without the need for electron generation and interaction or part destruction. a) shows the general arrangement of a high value component and an exemplar region specific microstructure being resolved into crystal types/orientation b) unlike traditional methods of materials characterisation SRAS makes use of surface acoustic waves to evaluate microstructures (d) by evaluating the surface wave velocity as a result of laser excitation c) selective etching of metal crystals can result in a facetted surface which is useful for characterisation this is exploited in DRM € and also 'Cryschem' (f). (d,e and f) are all published results on real materials illustrating the utility of these technologies.

learn more about their microstructure. Rayleigh would later identify the fundamental resolution limits which can be expected from visible light and so constraining optical microscopy and limiting what can be theoretically resolved.

Modern materials analysis techniques build upon the principles first identified over 100 years ago. The physics remains the same while our need for technologies, which give the same (or similar information) has changed dramatically. As our desire to confidently exploit the load bearing capacity of materials extends towards theoretical limits, practical solutions are required which draw upon measurands. These must be more easily obtained and allow inference of material properties which add value manufacturing chain through enriching the so called 'digital twin' [11]. This represents a tremendous value proposition to manufacturers of high value goods. While the use of polarized light technologies for textural analysis is now well explored even for complex materials [12,13] and is readily available in metallographic labs new technologies are needed in order to enable property informed manufacturing of high value metallic components.

Three emergent technologies of interest – The *purist* materials engineer may be driven by absolute measurement for which the 'electron' is indispensable but in many cases in the production setting a comparator or coarse evaluation to provide a 'go/no go' filter may be sufficient. To illustrate this, our perspective provides introductory insight into three techniques which are being explored by the authors for the purpose of providing rapid and facile evaluation of materials in the production setting. While these vary in terms of utility and technology readiness, we intend to communicate their basic operating principle and show how these may be employed with the purpose of supporting a digital manufacturing future. This is particularly relevant in the future manufacturing setting which will be more dependent upon automation and as such free from the inspection and evaluation technologies currently undertaken by humans.

Surface acoustic waves (SAWs) present a useful means by which to conduct non-destructive analysis for macro defects and microstructure analysis. The velocity of acoustic waves in crystals is understood to be dependent upon the vector relationship to orientation of the crystal and is also interrupted by discontinuities should as grain boundaries or localised strains (*see* Fig. 1b). A more comprehensive introduction has been in shared in the literature [14]. Through localised excitation of the surface of a crystal using a pulsed laser a surface acoustic wave can be created. The velocity of this wave can be evaluated in the plane of the surface through measurement of the SAWs [15]. Grains of the order of 0.1mm have been evaluated by this technique but consideration of instrument resolution is complex as useful information can be extracted as a comparator even when microstructure is finer than this.

In the case of spatially resolved acoustic spectroscopy (SRAS) the frequency response of a laser grating excitation is used [12] and this technique has been used to characterise a wide variety of materials such as titanium, nickel and iron alloys resulting from a variety of processing techniques including forging and additive manufacturing. While SRAS works best on polished surfaces it can be used on a variety of as manufactured surfaces such as additively printed, ground or machined surfaces [16] but this is more challenging and restricted to lower resolutions (and therefore larger grain sizes). SRAS can image the microstructure and determine the crystallographic orientation and in recent developments it has been demonstrated to be able to determine the single crystal stiffness matrix of the material [17]. Despite being a laser ultrasound technique the fluence of the incident laser energy required and resultant localised heating is quite modest and meaning that this is non-destructive in the majority of target materials.

Crystal response to anisotropic chemical and electrochemical etching (here termed 'Cryschem') varies depending upon grain boundary

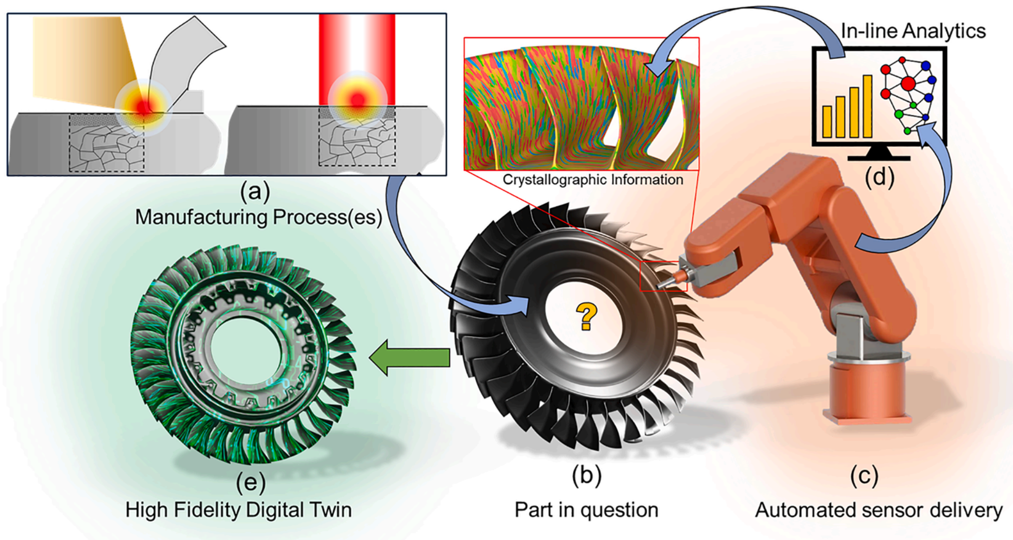

Fig. 2. A vision for metallurgical characterisation during production in additive manufacturing and allied high value processes. The 'integrity problem' persists as a blocker for the adoption of additive manufacturing in many sectors. The incorporation of non-invasive approaches for metals characterisation requires a rethink in industrial practice. The ability to move away from destructive and remote analysis to the direct evaluation of crystallography represents a significant prize for industrial practitioners. This approach also open the opportunity for integration with advanced automation technologies which can incorporate decision making capabilities to the advantage of the manufacturer.

density, grain orientation, and stress-strain condition. This is manifested as differential etching rates that can, under certain conditions, result in a faceted surface topography that is characteristic of the underlying crystal. Investigations show that automated material removal using an electrochemical jet (Cryschem) followed by direct surface topography measurement using interferometric metrology apparatus can coarsely identify and map dominant textures in cubic metallic specimens [18]. The mapping of local texture is limited by the capacity to measure enough etch facets per sampling division, where a statistically significant aggregation of etch facets is required for adequate texture characterisation.

Cryschem can prepare surfaces for metallographic inspection and provide crystallographic texture information at a modest spatial resolution in a rapid manner, at controlled depths [19]. It is insensitive to factory conditions and the optics required are widely in use in automated production processes. Integration of this technology to the production setting therefore does not require substantial adaption to existing factories, where processing and measurement are expected to occur in an in-line manner. The faceted topography that results from etching of polycrystalline surfaces is also known to affect the reflectivity of grains. It is this very same feature that first allowed to see and analyse the microstructure of solids using light microscopy in the early 1860s [20]. By measuring the direction and intensity of light as it is reflected at such facets, it is possible to reconstruct the local surface topography and, in turn, map the orientation of the underlying crystal lattice across the sample surface (Fig. 1e).

Building on this principle, directional reflectance microscopy (DRM) captures a series of optical micrographs from an etched crystalline under varying illumination angle [21]. However, this traditional information can be compiled into a much richer data set. The angle-dependent surface reflectivity is then arranged into "reflectance profiles" which may be analysed using either analytical or machine learning models (depending on the complexity of the signal) to compute crystallographic orientation [22,23]. DRM has been successfully employed on a variety of metals, metal alloys, and even ceramics and composite materials [24]. While requirements on sample surface finish are not stringent, orientation mapping by DRM relies on using etchants which yield surface faceting with consistent and known crystallography. Moreover, individual reflectance profiles must include contributions from multiple facets to ensure accurate measurements. This limits the spatial resolution of the technique and restricts DRM analysis to materials with average grain size of the order of several tens of microns.

A promising vision for the future - While the authors concede that the electron has an important part to play for some time to come in materials analysis there is a need to consider approaches which do not require a vacuum for digital manufacturing applications. However, competitive approaches are simply not sufficiently mature to meet widescale application at this time. The technologies considered here, DRM, SRAS and CrysChem all suffer from common limitations. For example, they all depend on optical resolution limits, require non-standard apparatus and the algorithms to 'solve' the crystallography remain limited to a small number of crystal classes. Where feature sizes are smaller than the fundamental resolution limits, it may be possible to characterise aggregated population effects to draw conclusions about certain aspects of the material. However, these approaches are still very much in their infancy. The authors hope that this brief introduction to the general arrangement and capability of these technique will enable other scholars to explore non-electron-based methods for evaluation of metals in the production setting. There are clear opportunities to enhance the solution and usability of these. A vision for industrial deployment for each of these in a future factory setting is provided below where processes of any description (illustrated by cutting and laser processing Fig. 2a) give rise to a high value component Fig. 2.b. It is perfectly reasonable to expect that such processes will be fully automated in the future and therefore entirely reasonable that the characterisation tools we will use should also be automated. Fig. 2c and 2d show illustrated the robotic delivery of an analytical tool such as SRAS/DRM/Cryschem and the data set which emerges. When recombining this data into a spatially registered format it is possible to conceive of a digital twin arising from this Fig. 2e. The emergence of 'big data' as a research field and how this will change materials science is largely a story in writing at the time of writing. However, it is immediately obvious that any such approach would be enriched by the provision of rapidly gathered data sources [25,26].

To progress these technologies towards adoption, engineering limitations must be overcome. Firstly, the current state-of-the-art allows all technologies to be deployed easily for more simple crystal primitives. Difficulties emerge where systems governed by more lattice parameters (leading to losses in symmetry) prevent solutions from being found.

However, comprehensive evaluation of microstructures should not be expected from these techniques nor, despite the title of this perspective, should they replace tried and tested approaches for detailed metallurgical analysis. Rather, these techniques are intended as complementary and informative of the material-process interaction at point of manufacture in a rapid manner. As such, with some a priori knowledge of the specimen in question valuable information can still be obtained as to the integrity of a multitude of alloy systems.

Secondly the technologies exhibited here will benefit from automation in due course in order to be integrated into the manufacturing process chains of the future. In this case automation extends beyond simply gathering data at the appropriate juncture but also using this information to inform downstream processes. This may include corrective/repair approaches for example [27] or inform heat treatment strategies to deliver so called 'digital materials' in which the properties of volumetric elements within a single component can be tailored. It is perhaps inevitable that, as our ability to gather and process data grows, the need for computational methods to utilise these will also become apparent. As such the field will rely on materials scientists, engineers and computer scientists to make the factory of the future a reality.

Beyond the current state-of-the-art we may wish to consider evaluating mechanical properties through such techniques. In the opinion of the authors this is perfectly plausible alongside data processing capabilities which will allow correlation at scale. Since compositional analysis is not currently possibly with these techniques (although one could imagine the integration of some form of spectroscopy) and resolution will always be limited (acknowledging Rayleigh once again) the need for there will continue to be a need for the electron and X-Ray based technologies for some time. Nonetheless, we would hope this note provides stimulus for parallel investigation.

Declaration of competing interest

The authors have declared no conflict of interest

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