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Sustainable Science Through a Case Study of Sample Preparation Using 3D Printed Tools.

By Lawrence Whitmore[1]

Abstract

The combination of cloud-based resources, user-friendly cloud-based design applications and 3D printing (3DP) is making possible a new sustainable paradigm in scientific research. Tools and components can be self-made by downloading model designs, optimizing those designs for individual experiments and printing locally. Together with the use of materials for 3DP filaments derived from renewable resources and recycling of old printed structures, science labs and institutes can significantly reduce their carbon footprint to meet their Sustainable Development Goals. This new sustainable paradigm is evaluated through a case study of sample preparation - a fundamental aspect of materials science. For high quality investigation of material microstructures, even to the atomic scale, sample preparation is critical. A range of 3DP plastic tools for preparing samples has been developed. The design and fabrication of a 3DP vibrational polishing machine and a 3DP dimpling machine are described along with test results from microstructural analyses of brass and silicon that show the high-quality scientific studies possible using these low-CO2e tools.

Keywords: 3D printing, sustainable development, sustainability mindset, carbon footprint, materials science, sample preparation.

1. Introduction

The nineteen-sixties and -seventies witnessed a rise in movements seeking change in social values and social structures around the world where human activities were felt to be unfair, unjust or otherwise not right (Gamson 1990; Hall 2008). As they grew, these movements of conscience begun to reach the minds of more people and led to changes in government policies and the development of organizations to implement change and improvement in societal practices (Cox 2018).

The environmental movement grew out of this era. Books such as *Silent Spring* (Carson 1962) and *The Limits to Growth* (Meadows 1972), examined more deeply the serious issues facing humanity in terms of our impact upon the Earth; in particular, deforestation through excessive logging, destruction of the seas through over-fishing and of the land through over-farming, massive pollution and the build-up of waste (industrial, household and of particular concern, radioactive waste). Movies such as *Soylent Green* (1973), *Logan's Run* (1976) and *The China Syndrome* (1979) brought these issues further into public awareness.

Although we can imagine sustainability as an issue only of the current time, it is one of the oldest issues that has faced our species: any population, whether it is a tribe, village, town or city, must live within the limits imposed on it by the ability of the land to provide food and process waste. However, while in earlier times community size was limited by the local

|[1]Department of Chemistry and Physics of Materials, Paris Lodron University of Salzburg, 5020-Salzburg.

environment, now the issue is global, and we are faced with not only exhausting the planets' capability of sustaining us but also of wiping out the other species that we share the planet with. According to environmental activist and winner of the 1997 Goldman Environmental Prize (www.goldmanprize.org) Terri Swearingen, 'we are living on this planet as if we had another one to go to'; but we don't.

Earth is the source of our sustenance, particularly the bio-sphere - the thin layer of organic life covering the surface (Raven 2022) (above and below water). The natural processes that sustain the bio-sphere are deeply integrated and finely balanced (Benton 1995), resembling in many ways those of a living being (Lovelock 1972). When we extract too much through farming, fishing, logging and mining, we cause imbalance and exhaust the bio-sphere (Holling 1973); and when we pollute, we damage it directly (Taubert etc. 2018; Ceballos etc. 2002; Ceballos etc. 2015; Pimm etc. 2000). Such over-use is ultimately an act of suicide as we kill off our own source of sustenance and nourishment (Lovelock 2009).

In 1992, the United Nations Conference on Environment & Development (the Earth Summit) held in Rio de Janeiro sought to address these issues through global initiative: AGENDA 21 was formulated as a protocol for reducing the impact of human activities upon the environment (Agenda 21, 1992). The report outlined action plans for regulating our activities through nine different groups or sectors under the umbrella heading of sustainable development.

Section III-31 of the report detailed how the scientific and technological community would be developed as well as the role it would play in developing new more sustainable methods for other sectors and in informing the public about the key principles and facts at play. Progress has been reported and protocols updated in subsequent meetings, the most recent being the UN Sustainable Development Summit in 2015, in which AGENDA 30 and the 17 Sustainable Development Goals were formulated (The 17 Goals, 2015).

In the late 1990's the concept of an ecological footprint was conceived as a means of quantifying human impact upon the environment (van den Bergh etc. 1999; Wackernackel etc. 1996). This was later reduced to the carbon footprint (Hertwich etc. 2009), a catch-all parameter that can be calculated to estimate our impact. According to the Encyclopedia Brittanica a carbon footprint is the amount of carbon dioxide (CO_2) emissions associated with all the activities of a person or entity and often includes the emissions of other greenhouse gases (Selin 2023), generally converted into carbon dioxide equivalent mass (CO2e) for comparison (measured in grams or kilograms). Sustainable development is then interpreted as the reduction in carbon footprint of human activities in different categories such as economic sector or geographical area. Activities with positive CO2e, such as air-travel, need to be reduced or made more efficient, while activities with low or negative CO2e, such as recycling and use of renewable energy sources, need to be developed and increased.

CO2e can be calculated for commodities and activities based upon the relevant emission factors (EF) using Equation 1, where activity or commodity is a quantification of the activity or commodity (such as duration of an activity, mass of material or amount of energy used). Specific EFs are derived from detailed life-cycle studies and are available in online databases, while online calculators can be used to estimate the carbon footprint of individuals and organizations (Carbon Footprint Country Specific Electricity Grid Greenhouse Gas Emission Factors 2021; Calculate Your Carbon Footprint 2023).

$$\text{Equivalent carbon (CO2e)} = \text{(activity or commodity)} \times \text{Emission Factor (EF)} \qquad \text{Eq. 1.}$$

A low-carbon sustainable paradigm in science is now emerging where cloud computing and 3D printing (3DP) technologies allow for designs for tools and components to be downloaded, optimized for individual experiments and printed locally. This reduces the very large CO2e associated with mining, refinement and distribution of raw materials, the complicated energy-consuming manufacturing processes, and the packing and shipping of materials and finished products. It also facilitates science and technology in a struggling economic environment and with a failing supply chain, where advanced equipment might not be readily available, and would benefit the scientific environment where innovation would have great potential to improve experimental procedures in economical and sustainable ways.

Successful implementation requires a sustainability mindset in the management of research labs and in the design of projects and experiments, and the willingness to innovate or support innovation of new designs and methods. Only in the case of complex specialized and electrical equipment, need tools be sourced externally; and even then, the new modular approach to electrical hardware (e.g. the Arduino system) can facilitate on-site fabrication of more complex devices.

The current study explores this new paradigm through a case study of sample preparation for microstructural analysis, a task that is at the foundation of materials science. Its contention is that on-site production of tools and components using 3DP can reduce the carbon footprint of research groups and science laboratories helping them meet their sustainable development goals; particularly in comparison to the large and heavy power-greedy and consumables-greedy machines that were designed and manufactured before the sustainable era (Bass 2007) without consideration for sustainability. However, this is only so when low-carbon print media and appropriate recycling are used (Jorgenson 2019; Letcher 2020; Worrell 2014).

Microstructural analysis is an aspect of materials science that reveals the structure of materials at the scale of microns and nanometers (McCall etc. 1973). It is at this scale that atoms bind into regular crystalline structures, which form the basis of bulk materials and their properties such as density, strength and ductility. Methods of microstructural analysis, such as electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM), require highly specialized methods of sample preparation, which in turn require highly specialized tools.

The following sections examine in more detail the principles of sustainability (section 2) together with a case study of sample preparation for microstructural analysis (section 3). Two tools for sample preparation are designed using an online design platform and printed using a consumer grade 3D printer. These tools are then evaluated by preparing and analyzing samples with EBSD and TEM.

2. Sustainability and science

A visionary of the early 20th century said that 'before it is too late, humankind must appeal to new thinking habits, new mindsets and new schools of thought' (Steiner

1919): our mind-set at the time had led us into the disaster of the first world war. This insight was reiterated in *The Limits to Growth* [Meadows 1972] by the observation that the planet can sustain a population of responsible human beings but not of greedy egoists: The world system is simply not ample enough nor generous enough to accommodate much longer such egocentric and conflictive behavior by its inhabitants (p192). These observations are still applicable today, and perhaps even more so, as our population has grown by around 110 % since the 1970's and still lacks a consistent responsible attitude regarding our relationship with the planet.

A mindset is a collection of views, beliefs and attitudes that governs our thinking, decision making and actions [Burnette etc. 2013; Dweck 2017]. These factors are closely interconnected, so that a change of attitude tends to be possible only when the associated views and beliefs are strong enough or urgent enough to overcome the resistance of existing habitual attitudes. Philosophical studies have often cited pride and greed as key elements in man's downfall (Alighieri 1265-1321; Antonetti etc. 2014): while greed drives us to over-consume, pride keeps us from questioning our actions or of considering their effect upon others and upon the planet itself (Barnett etc. 2005). This means that change is in many ways a fight against ourselves and our own weaknesses and negative tendencies, so that sustainable development can be considered not only in terms of technological development but also in terms of self-development.

To bring about a sustainable society, a new sustainability mindset is called for (Hermes etc. 2018; Kelley etc. 2014): one that appreciates the key issues of sustainability, acknowledges the need for a rapid and systemic response and is prepared to make the effort. The growth of such a mindset, through education, action programs and initiatives, could be considered an evolutionary step forward as we manage and regulate our own behavior to protect the environment (Going Green 2023; Segovia 2010) and preserve ourselves from extinction (Wilson 2003; Barnosky etc. 2011; Raup 1986; Chure 2022). With sustainability at the forefront of our thinking we can discover and innovate new ways and methods that allow us to live in greater harmony with the planet for our mutual benefit (Doak etc. 2014; Eggermont etc. 2015; Kareiva etc. 2012) and that save our planet for future generations of humans. Native peoples have often spoken about living with the planet rather than living off the planet, which demonstrates an integrated win-win relationship with the Earth based on stewardship rather than parasitism (Ashby etc. 2017; Worrell etc. 2016; International Commission on the Futures of Education 2021).

From as early as the C16th innovation meant novel change, experimental variation and alteration of established practices [Innovation 2023]. In practice, innovation works by considering the question: how can it be done better? It re-examines problems in the light of new advances in knowledge, and connects the dots between existing technologies to bring about incremental update or upgrade of existing solutions. As an example, man at one time invented the bridge, but later improved the bridge by making it lighter and stronger through innovation and the use of new materials.

Presently, advances in digital technology are changing our world, with potential for many sustainable improvements. The development of high-speed internet and its availability worldwide, combined with the data-processing capabilities of 10 nm line-width integrated circuits (Heterogeneous Integration Roadmap, 2023) constitutes a platform of data processing and exchange that underpins the digital revolution (Shalf etc. 2015; Moore

1965). Instead of data being stored and processed locally on individual personal computers, it is stored remotely and processed remotely – in the cloud (Hutchinson etc. 2009; Buyya etc. 2009; Wang 2010; Zhang etc. 2010). Devices such as personal computers, smart phones and the *Internet of Things* in general (Diaz etc. 2016; Yelick et al 2011) then access the cloud via high-speed routers to transmit, receive and share data. Remote servers and applications have the capacity and power to store and process vast amounts of data and large numbers of requests simultaneously. A wide range of cloud-based applications designed for the scientific community (Rehr etc. 2010; Foster 2017) include *Materials Cloud* (Materials Cloud 2023), the *Open Science Data Cloud* (Open Science Data Cloud 2023), the *European Open Science Cloud* (European Open Science Cloud 2023) and the *China Science and Technology Cloud* (China Science and Technology Cloud 2023). Cloud resources for productivity and design include most common Office suites, graphics packages such as Photoshop and free applications such as Tinkercad.

The relatively recent technology of 3D printing likewise has great potential for change towards sustainability in the area of construction. As a form of additive manufacture (AM) it builds 3D structures layer by layer from a digital model (Shahrubudin etc. 2019; Ngo etc. 2018). Complex structures can be produced with significantly less waste material than traditional milling or sculpting that hew structures from a solid work piece. With use of renewable sourced biodegradable print media combined with either recycling or proper biodegradation, 3DP has great potential for building structures in a sustainable society.

There are several methods of 3D printing, but the two most common are material extrusion and vat polymerization. Material extrusion builds a 3D structure by pressing a print medium out of a nozzle (print head), while vat polymerization slowly draws a 3D structure out from a liquid bath or vat while a UV laser beam polymerizes or hardens the liquid layer by layer. A consumer-level printer costing as little as €250 can print up to 10 g per hour and achieve dimensional tolerances better than 100 μm in prints as large as 300 mm and surface roughness better than 20 μm (Hartcher-O'Brien etc. 2019; Islam etc. 2014). The typical power requirement of a table-top 3D printer is 250 W, which means that a 4-hour print will use 1 kWh of electricity, roughly equal to €0.12 at today's rates and approx. 250 g of CO2e per kWh of electricity based on an EF of 250 g CO2e per kWh for electricity in Europe (Greenhouse Gas Emission Intensity of Electricity Generation in Europe, 2023) and calculated using Equation 1.

The printer used in this study was a Creality Ender 3 V2, which uses a version of material extrusion known as fused deposition modelling (FDM) together with a thin plastic filament. The filament used in this study was polylactic acid (PLA), a bioplastic derived from renewable resources such as corn or sugar cane (Doane 1992) that is biodegradable (Rajeshkumar etc. 2021; Swetar 2023; Xu etc. 2022; Gebler etc. 2014) and forms structures with mechanical properties suitable for a wide range of practical applications (Scaffaro etc. 2018). PLA can be recycled using a shredder that breaks up old structures and an extruder that melts the pieces and produces new filament (Aurus etc. 2010).

PLA has one of the lowest associated carbon emissions of any plastic based on its life-cycle, with an EF of approx. 0.6 kg CO2 per kg (Morao etc. 2019; Vink etc. 2003). If a 4 h print uses 40 g of PLA, then the CO2e would be roughly equal to 25 g, using Equation 1, which combined with 250 g of CO2e for electricity would be a total of roughly 275 g of CO2e for the print.

However, with 3D printing entering the consumer market a new set of problems has arisen. In particular is the excessive use of plastics driven by irresponsible use of the technology and the subsequent increase in pollution. Not all filaments are sourced from renewable resources and not all filaments are biodegradable. As is the case with all new technologies, at some point they will likely need regulating. Various ways to regulate 3D printing include connecting 3D printers to an internet account that tracks and controls print volume, the printing of an ID on the underside of prints (as is currently in use with many laser printers) and taxation of filaments based on their sustainability.

Therefore, to work with 3DP in a sustainable way, the following principles should be applied:

- do not print unnecessarily;
- use a biodegradable filament obtained from renewable sources, such as PLA;
- print low-quality low-density test prints when developing new models;
- use lowest possible infill according to the strength requirements of the structure;
- minimize support material by designing beveled undersides rather than flat;
- design holes and recesses into parts to reduce filament requirements;
- tap threads directly rather than using metal inserts (to facilitate recycling);
- recycle old printed structures.

3. Case study: sample preparation for microstructural analysis

Materials science - the study of the nature and properties of materials - is made through testing and analyzing samples. A principal at the foundation of materials science is that macroscopic properties, such as density, strength and ductility, are derived from structures at the micron and sub-micron scales, which in turn are derived from the forces and symmetries at the atomic scale (Whitmore etc. 2019). Therefore, a significant fraction of materials science is devoted to the study of microstructures using various forms of microscopy and related analytical techniques.

EBSD is a technique that uses an electron beam to analyze the phase and orientation of grains with a typical spatial resolution of 0.1-1 μm. This method requires that surfaces are prepared to extremely high standard so that the uppermost layer of approx. 100 nm (1/10th of a micron) is free from contamination and preparation-induced damage. Grinding and polishing using increasingly fine abrasives are required and a final stage of extremely fine vibrational polishing. This case study presents a vibrational polishing machine produced using 3DP (Whitmore 2023) along with previously unpublished results. Smaller micro-structures down to approx. 0.1 nm in size are examined using TEM, which demands even more stringent sample preparation. Samples must be thinned to less than 100 nm thickness so that an electron beam, with typically 200 kV energy, might pass through and form an image of microstructures inside the sample on an imaging screen or sensor. In addition to normal grinding and polishing, a dimpling machine is used to form a concave impression in the sample so that the thin central area can then be thinned using a high energy ion beam to the required thickness. This case study presents a dimpling machine produced using 3DP (Whitmore 2023) along with previously unpublished results.

	Vibrational polishing machine	Dimple grinder/polisher
Filament type	PLA	PLA
Layer thickness, mm	0.2	0.2
Infill density, %	15	15
Print speed (first layer), mm/s	20	20
Print speed (bulk), mm/s	50	50
Wall thickness, mm	1	1
Filament mass, g (€)	120	160
Print time, h	18	20
Energy consumption, kWh (€)	4.5	5
Combined printing cost, €	6	7
Carbon equivalent (CO2e), kg	1.2	1.35

Table 1. Quantities used for 3D printing the devices, energy and filament consumption, cost and equivalent carbon emissions.

Table 1 shows details of the 3D printing for the two devices. Infill density and wall width were chosen to optimize the strength and weight of the structures, while layer height and print speed were chosen to optimize surface quality and dimensional accuracy. Energy consumption is based on the 250 Wh printer and printing cost combines electricity at the current rate of €0.12 per kWh and cost of PLA filament at €40 per kg. The carbon equivalent is calculated using Equation 1 and combining both energy consumption and filament usage.

In both cases, commercial machines are available. However, they are designed and built on a historical premise that did not take sustainability into account. A typical commercial vibrational polishing machine weighs in excess of 80 kg (500 times heavier than the 3DP plastic model presented here) and has considerable power and consumable requirements. Complex metal constructions have very large carbon footprints due to the life-cycle of mining, smelting, shipping, machining, welding, painting and disposal. The volume of polishing fluid required in a commercial polisher is around 10 times more than in the 3DP machine and the polishing cloth is ten times larger and cannot be reused once removed, whereas the 3DP machine can be reused multiple times. The CO2e of shipping machinery is also very large with an EF of around 15 g CO2e per tonne-km for sea freight and 2 kg CO2e per tonne-km for air-freight (Freight-Shipping 2023).

3.1. Vibrational polishing machine

Vibrational polishing is used as a final stage in preparing surfaces for metallographic analysis and advanced analytical methods such as EBSD (Dingley etc. 2018; Humphreys 2001; Voort etc. 2006; Nowell etc. 2005) that determines grain phase and orientation. The gentle effect of vibration combined with a suspension of sub-micron abrasive particles in water produces a very flat and smooth surface finish with little or no preparation-induced damage.

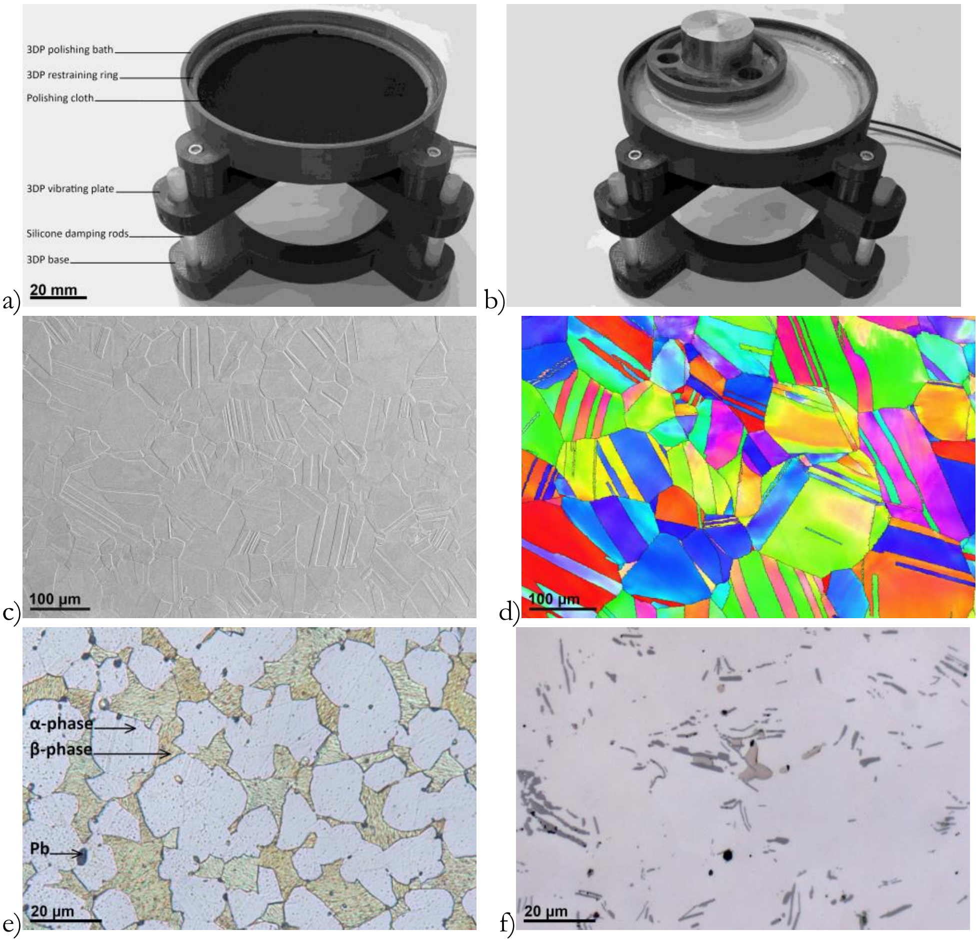


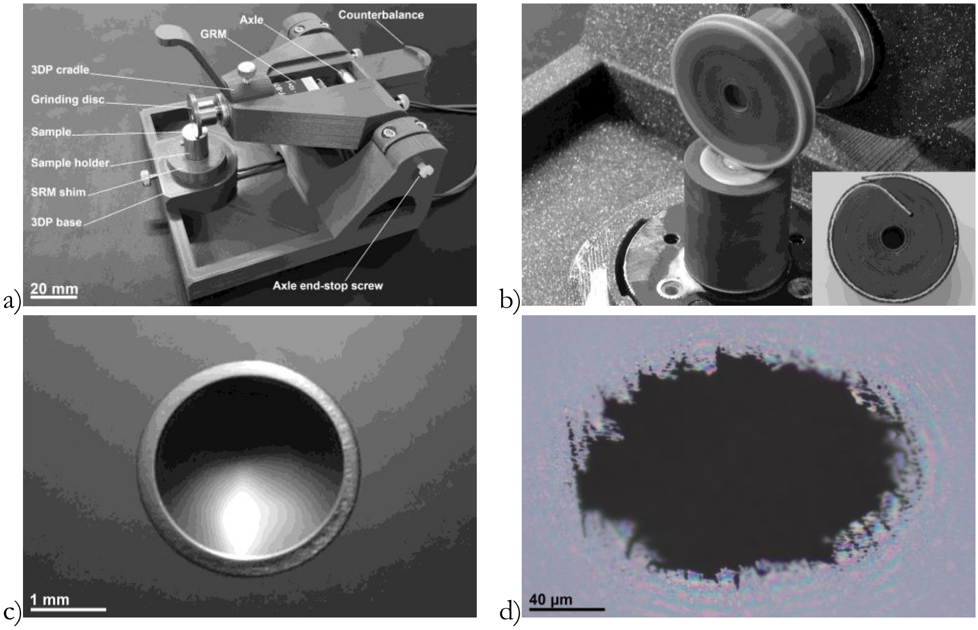
Figure 1. The 3DP vibrational polishing machine a) with key features indicated, b) with samples and polishing solution, and different samples prepared using the device: c) a single-phase brass showing grains and twins (lamella structures) within grains, d) a grain orientation map of the same where color represents lattice orientation, e) a duplex brass showing the α- and β- phases and f) intermetallic precipitates in aluminum 6060 alloy.

Figure 1 shows the vibrational polishing machine constructed using 3DP. The device was designed to polish samples up to 2 cm in size and consisted of three 3DP plastic components - a base, a vibrating plate and a bath – that required 120 g of PLA filament and 18 hours to print (using high quality print settings). The base was optimized for stability, while the vibrating plate and bath were optimized for lightness. The entire device required no support material when printing, and therefore eliminated filament wastage. A €5 DC motor was used with an offset load or eccentric rotating mass (ERM) to generate vibration in the horizontal plane, and was fixed centrally in the 3DP vibrating plate directly underneath the polishing bath. Silicone cord was used for the four damping columns. Figure 1 b) shows the machine in use where three samples are inserted into a 3DP sample holder and a stainless-steel cylinder is used to apply a load onto the samples.

Figure 1 c) shows a single-phase brass after polishing and etching and d) a grain orientation map made using EBSD. Different colors (or shades in printed version) in the map represent different orientations of the base copper micro-structure; this is interpreted using a color code (not shown) that relates color to lattice orientation relative to the sample surface. Figure 1 e) is a two-phase brass after etching, showing grain size and phase fraction. The dark spots are lead particles (precipitates) that improve the machinability of the brass. Figure 1 f) is an aluminum 6060 sample directly after vibrational polishing and without etching showing intermetallic precipitates. In each case the surface is almost perfect for this type of analysis showing no obvious scratches from preparation, and it is clear that using the 3DP machine very high-quality surface preparation is possible.

3.2. Dimple grinder-polisher

Dimple grinding-polishing is used as an intermediate stage in preparing samples for TEM (Goodhew 1984; Williams etc. 2009). The purpose of the dimpler is to form a spherical impression in a 3 mm diameter round sample so that the central area reaches a thickness of 10-15 μm while maintaining a thicker outer edge for support. A subsequent stage of ion-beam milling thins a central hole so that the material around the edge of the hole is electron transparent (i.e. less than 100 nm thick).



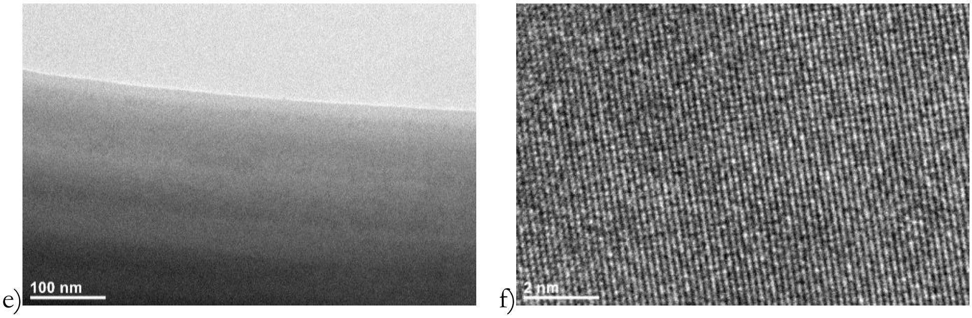


Figure 2. The 3DP dimpler a) with main features indicated, b) with detail of the 3DP polishing disc and sample holder with inset detail of cloth strip mounted on the plastic disc, and results from a silicon sample showing c) polished dimple, d) ion milling showing the thinned hole, e) TEM image showing the high-quality defect-free sample and f) image of the silicon lattice at the [001] axis with bright spots representing silicon atoms.

Figure 2 shows the dimpler constructed using 3DP. The device consists of two main 3DP plastic components - a base and a cradle – requiring a total of 160 g of filament and 20 hours to print (using high quality print settings). Two DC motors are used for rotation of the sample (the SRM) and rotation of the grinding/polishing disc (the GRM), the actions of which in combination produce the dimple. A stainless-steel or ceramic rod was used as a low-friction axle for the cradle. The design is optimized for stiffness and stability using a block-type construction and recesses to save filament but also to house batteries if used as a power source. Less than 5 % of filament mass was used for disposable support material. Figure 2 b) shows details of the dimple grinding process, and the inset image shows a 3DP plastic polishing disc with a strip of polishing cloth attached using silicone adhesive for polishing inside the dimple.

Figure 2 c) shows a silicon sample after dimpling and d) the central area after ion-beam milling. The sample at the hole edge is less than 100 nm thick so that the electron beam can pass through. Figure 2 e) shows an image formed by the electron beam, where the silicon is free from any obvious preparation-induced defects. Figure 2 f) shows the arrangement of atoms in the silicon sample at the [001] crystallographic axis and the spacing between atoms is 0.192 nm. Such high-resolution study is only possible with very best sample preparation, indicating that the 3DP dimpler is suitable for this type of investigation.

4. Summary and conclusion

Sustainable development requires us to rethink how we go about our lives so that we minimize our impact upon the planet that sustains us and particularly upon the delicate and finely balanced bio-sphere. At issue is the wellbeing of the planet and our own survival on it. In the context of scientific research, it has been shown that 3D printing can be used to produce tools and components suitable for preparing samples for high quality scientific investigation of microstructures. Both EBSD and TEM are methods of analysis that require the most stringent sample preparation, and in both cases, it is shown that high quality samples can be produced using 3DP plastic tools prepared using a consumer-grade 3D printer. This contends that a substantial fraction of tools and components used in science and technology can be prepared in this way and recycled when no longer required to reduce the overall carbon footprint of scientific labs.

The samples analyzed here (brass, silicon and aluminum 6060 alloy) are important materials used across a number of industries including automotive, aeronautical and semiconductor. The analysis of grains is used in materials science to study the relationship between macroscopic properties and the microstructure, and to engineer specific properties into materials such as metal alloys. The size of grains, and the size and concentration of precipitates and deformation structures such as twins, all influence the mechanical properties of bulk material. It is for this reason that sample preparation should not leave traces of its own in the sample otherwise this would confuse the analysis. In particular, the absence of scratches in the surface and the cleanliness of the surfaces is a measure of the quality of sample preparation achieved using the 3DP plastic tools.

Ultimately sustainable development comes down to individuals and institutes making the effort to think and act sustainably. Estimations of carbon footprint can be explicitly included in the design of projects and experiments (each experiment will have an own carbon footprint) and day-to-day operations of labs and institutes. Labs can equally have a carbon budget as they can a financial budget, and new equipment can be assessed in terms of its CO2e before acquisition.

Although devices prepared with 3D printing are not expected to be as durable as commercial tools, and should not be considered as competition, they have many sustainable and scientific advantages: they can be tailor-made to fit the exact requirements of experiments, can be recycled and re-printed, are biodegradable, can be sourced from renewable resources, can be self-made within a few hours, require no shipping, cost only a few Euros to print and have very low power consumption. And it has been shown here that such 3D printed plastic tools can indeed be used to prepare samples to the highest standards required for high quality analytical studies.

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