



Review of the Validity of the Use of Artificial Specimens for Characterizing the Mechanical Properties of Rocks

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Abstract

The field of rock mechanics is concerned with the response of rocks to the forces acting on them, characterizing this behaviour in specific environments and under varying loading conditions. This review discusses the suitability of current mechanical testing methods for inhomogeneous rock bodies, with a specific focus on the use of artificial samples in place of real rocks in these tests. The use of artificial materials such as cement, resins, and sand-based mixtures is reviewed, as is the manufacture of three-dimensionally printed samples. The benefits and drawbacks of using such specimens in mechanical tests, and the validity of their simulation of real rock are discussed. There is evidence that 3D-printed samples have the advantage of overcoming the problems of specimen reproducibility and also make possible the ability to test the response of specific defects to loading. There is thus great potential for the use of 3D printing in this field. However, the review concludes that further post-processing and careful thought about the materials used must be carried out to ensure that printed samples are of adequate strength and brittleness to accurately simulate real rocks.

Keywords Rock simulant · 3D printing · Fracture · Blasting

1 Introduction

To be able to model the response of rocks to dynamic and quasistatic loading such as occurs in earthquakes, mining or quarry blasting, the mechanical and fracture behaviour of rocks must be investigated, including at high rates of loading (Green and Perkins 1968; Stimpson 1970; Brace and Jones 1971). In addition, the testing methods used must cover a wide range of environments and loading mechanisms (Zhu et al. 2018b). It must be kept in mind that rocks have a wide range of chemical compositions, microstructures, and crack patterns, all of which affect their mechanical response. Current quasistatic and dynamic materials testing methods must therefore be assessed both for their ability to measure the properties of rocks adequately and to identify where new methods are needed to overcome current limitations.

There has been interest in using artificial model materials for as long as the mechanical testing of rocks has been performed (Stimpson 1970). When using a simulant material, it

is important that the validity of using a simulant is assessed carefully so that the property of the ‘real’ material that is being assessed is replicated adequately. It is not necessary to replicate all aspects of the material, but similarity in certain parameters is essential, as otherwise the conclusions reached cannot reliably be used to make predictions about the behaviour one might expect in the rocks themselves. A simulant with poor predictive capability is not much of a simulant. A problem in many current investigations is that the mechanical properties of simulant materials are not similar enough to real rocks (Wong and Chau 1998; Jiang and Zhao 2015; Jiang et al. 2016b). It is our contention that there does not seem to be sufficient justification in the published literature that the discrepancies between the mechanical properties of rocks and rock simulants is being addressed or can be overcome so as to provide true predictive capability.

However, there are various arguments going back 100 years or so that support the use of non-perfect material simulants. In particular there is a long tradition of making models out of optically transparent (but mechanically weak) materials to obtain insights into both the internal stress fields (Coker 1910, 1912; Coker and Donaldson 1913) and the fracture processes (Kolsky and Shearman 1949; Kolsky and Rader 1968; Field and Ladegaard-Pedersen 1971) that

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occur in much stronger materials. The reason is that nearly all load-bearing materials of interest, whether natural or artificial, are opaque to visible light. This is not a coincidence (Gilman 2003), but it does make measuring what is going on inside a real loaded structure problematic. Surface measurements of displacement are, of course, possible for opaque materials such as rocks (Zhang and Zhao 2014).

The physical phenomenon that allows the visualization of bulk stress fields is photoelasticity (Coker and Filon 1931; Frocht 1941, 1948). This phenomenon was first reported in silicate glass (and other transparent materials) by David Brewster early in the nineteenth century (Brewster 1815, 1816). The main problem with using silicate glasses to gain insight into internal stress fields is that their photoelastic constants are small (Filon 1902, 1910). Glass is also very brittle so that high loads and large strains cannot be investigated using this material (Mesnager 1913; Baud 1929; Favre 1930). However, around the beginning of the twentieth century a transparent polymer was developed that has a large optical sensitivity to stress. Very soon beautiful and striking images were being published of stress fields inside standard test specimens as well as small-scale models of structures loaded in various ways (Fig. 1).

A classic example of the utility of simulated specimens is shown in the photographs in Fig. 2 taken by Persson (1970). In this instance the aim was not to show that the material was equivalent to rock (and therefore predictions could be made about material performance), but to demonstrate some of the geometric and fragmentation effects of changing the detonation pressure in a blasting environment. The exact fracture pattern or fragment size distribution is not important, but the gross effects of diluting the charge can be seen: a reduction in the pressure (Fig. 2b) causes the zone of intense plastic deformation (akin to comminution in the rock blasting case) around the borehole (Fig. 2a) to disappear and the ring of shear fractures encroaches right up to the borehole. More information about these experiments may be found in (Field and Ladegaard-Pedersen 1971, 1972).

The fabrication of synthetic rocks using gypsum, for example, has been of interest in studies of the permeability of rocks to fluids (Ishutov et al. 2015, 2018; Yin et al. 2017; Kong et al. 2018b). In such studies, the mechanical strength of the simulant is not an issue: all that is required is that the synthetic rock has the same internal geometrical features and that the chemical nature of the surface that the fluid is flowing over has no effect. Such techniques have also been applied to the study of the effects of defects on the mechanical properties of rocks (Singh et al. 2015; Vogler et al. 2017; Kong et al. 2018a).

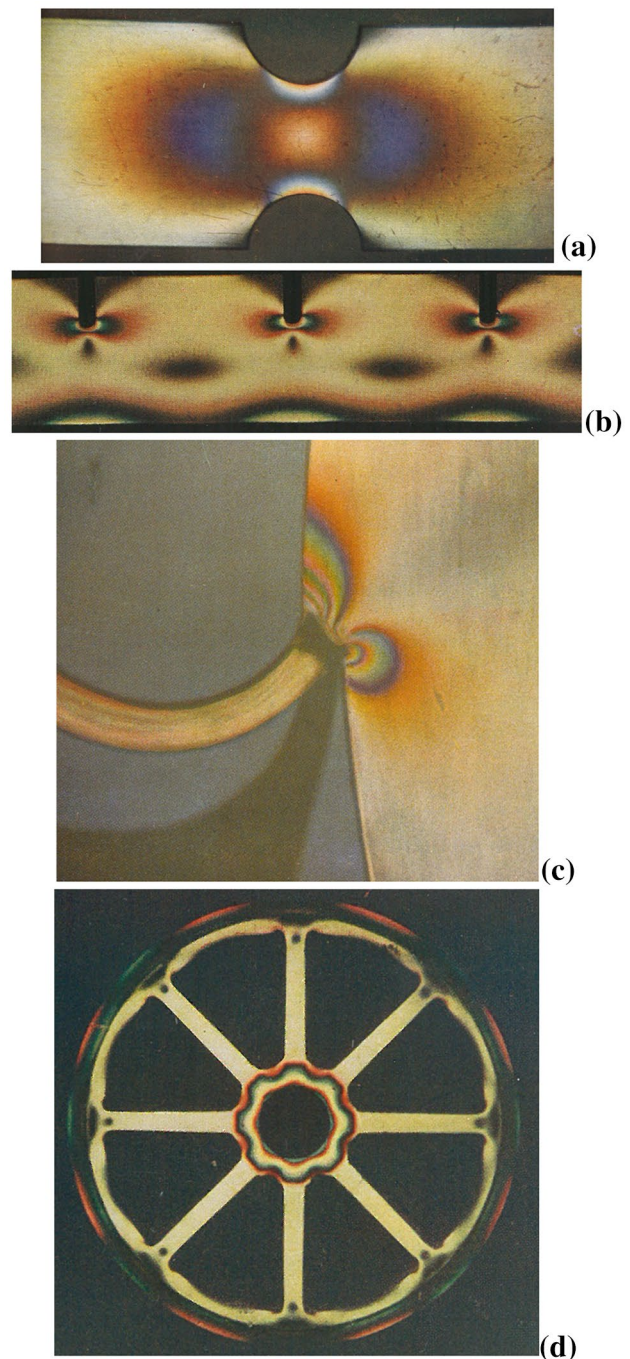
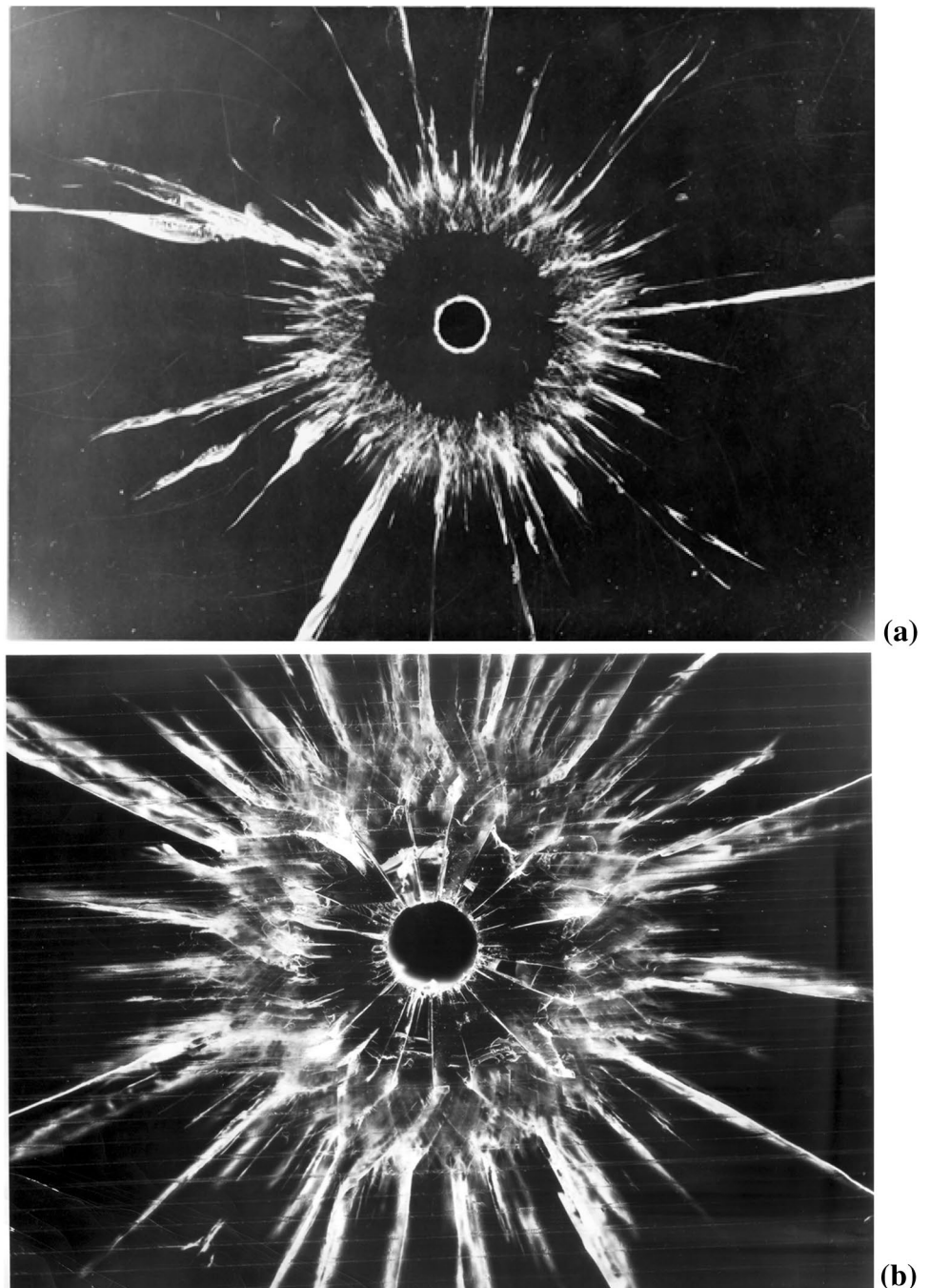


Fig. 1 A selection of colour photoelastic images from Coker and Filon's 1931 'Treatise on Photoelasticity'. **a** A loaded round-notched tensile specimen; **b** stress field around a linear array of cracks; **c** stress fields produced in a material during machining; **d** a model of a railway locomotive wheel (Coker and Filon 1931)

Fig. 2 Cross section of two boreholes in PMMA fired with different densities of PETN. In (a) a higher density of explosive increases the pressure seen in the surrounding plastic causing intense plastic deformation. Reducing the pressure as in (b) causes the ring of shear failures to encroach closer to the borehole. Width of view 10 cm (Persson 1970)



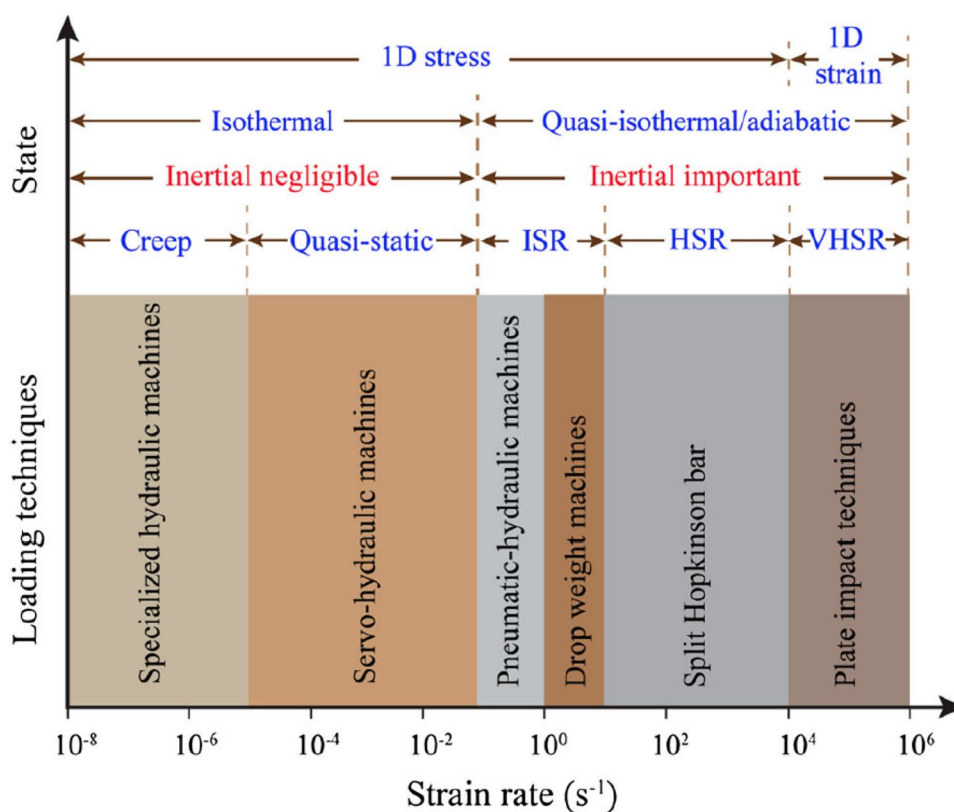
2 Uses and Limitations of Natural Rock Samples

As for other materials, the mechanical properties of rocks depend on the rate at which the load is applied (Field and Walley 2013; Zhang and Zhao 2014). Currently, techniques exist that cover strain rates ranging from 10^{-5} to 10^6 s^{-1} . The methods include dropweight machines, split Hopkinson pressure bars and shock-loading techniques. These are discussed in critical reviews by Field et al. (2004), Field

and Walley (2013) and are summarized in Fig. 3 (Zhang and Zhao 2014). The papers discussed in the review by (Zhang and Zhao 2014) primarily used natural rock specimens in their investigations, the exception being where photoelastic polymer models were used to study stress fields in rocks loaded in various ways (Gomez et al. 2002).

While results from the various testing methods mentioned above have resulted in important conclusions in various areas of rock mechanics, the use of specimens of natural rock in mechanical tests presents two major difficulties:

Fig. 3 A chart displaying the loading techniques used to study the dynamic mechanical properties of rocks at different strain rates. Those with a rate of approximately 10 s^{-1} and higher have to be studied using specialist experimental methods (Zhang and Zhao 2014)



1. It can be extremely difficult to find natural rock samples with the feature present that it is desired to test the effect of. Take cracks, for example: understanding the effect of these discontinuities on rock mechanical properties is crucial for many real-life applications, but it is difficult to find samples with the required crack network already present. Cracks can, of course, be made artificially in rock specimens before testing, and this technique is one that has been developed over the years e.g., (Amalokwu et al. 2015; Haeri et al. 2015; Roy et al. 2016). Originally this required methods such as the use of a wire saw (Dai et al. 2011) to create cracks in the rock material, allowing the mechanical response to loading around these features to be investigated. However, these processes are laborious and often led to cracks being produced with uncontrolled lengths or widths. More advanced methods (such as using a submillimeter diameter water jet) allow flaws to be created reproducibly (Lee and Jeon 2011). However, the cracks produced using this method had a rounded tip due to the water jet creating a larger hole at the point where it penetrated the specimen. Since the propagation and coalescence of cracks is well known to be affected by their geometry (Aliha et al. 2010; Khani et al. 2013; Kolvin et al. 2015; Sanyal et al. 2015), this is a clear limitation of this method.
2. Natural rock has a high degree of variability, making it difficult to generate consistent results from tests that

need a number of different samples even if they are taken from the same rock body. In addition, rocks normally being brittle materials, specimens are usually destroyed in most mechanical tests (Wu et al. 2016), meaning that repeat tests or investigation of other properties cannot be carried out.

To overcome these problems, the testing of more or less realistic artificial rock materials has become a major topic of research in rock mechanics (Ivars et al. 2011; Smith et al. 2014). The majority of this review will focus on the advances made in this field, exploring the advantages and limitations of the use of artificial samples, and discussing whether the current methods can sufficiently accurately simulate real rocks to be able to provide meaningful predictive conclusions.

3 Artificial Rock Materials

Artificial rock samples are created either by filling moulds or by 3D printing using a material chosen to produce a rock-simulating specimen. The main advantages of artificial rock materials is that the specimens are more homogeneous, cracks can be introduced of known position and geometry and they are cheaper (Fedrizzi et al. 2018). An investigation into crack growth showed that cracks could be created in

samples made from polyester resin, polymethylmethacrylate (PMMA), glass and cement (Dyskin et al. 2003). The cracks were made by embedding inclusions during specimen preparation in the mould. This technique is a more effective method than those used to create features in natural rock samples as many identical samples can be produced. However, all the materials used in Dyskin et al.'s investigation were homogeneous with respect to the scale of the crack, which real rock materials are not. This means that the effect on the mechanics of structural elements found in real heterogeneous rocks cannot be investigated using their method. This problem was also identified in an earlier investigation into the brittle fracture of rock using PMMA (Germanovich et al. 1994). Germanovich et al. concluded that propagation of 3D cracks results in failure of the samples they studied, but they noted that their specimens behaved very differently to real rocks due to differences in fabric, scale and extent of initial damage. These observations indicate the need to more accurately recreate the internal structure of rocks in artificial specimens. It is possible that more homogeneous rock materials might be better simulated by such a method, but that would require further investigation.

An investigation into the behaviour of multiple parallel cracks in Brazilian disc cement samples clearly displayed the benefit of being able to repeat tests and hence check the reproducibility of results when using artificial samples (Haeri et al. 2015). High reproducibility between three specimens was achieved for each crack combination. Figure 4 shows the breaking load of cracked specimens normalized by the average breaking load of uncracked specimens of the same material. This plot indicates that the three model specimens displayed a very similar normalized breaking load for the same number of cracks. Such reliable conclusions regarding the effect of cracks on load would be unattainable with real rock samples due to their heterogeneity.

Hence a technique like this allows a single variable to be investigated independently of other factors. However, there are difficulties in extending these conclusions to real rocks since other factors are likely to have an effect. So such an approach needs to be validated by the investigation of real rock samples.

While the use of artificial samples can clearly result in repeatable investigations and also allow the independent study of known defects, the properties of model materials must also be considered. (Wong and Chau 1998) created samples made from a mixture of barite, sands, plaster and water to mimic the behaviour of sandstone undergoing crack coalescence. Figure 5 shows a comparison of the stress–strain curves of the modelling material and sandstone. Wong and Chau concluded that the sharp drop in the curve shows that their model material and sandstone had similar brittle characteristics.

However, it should be noted that the plot for their model material is scaled up significantly, as they quote a value for its true Uniaxial Compressive Strength (UCS) as 2.09 MPa compared to around 100 MPa for sandstone. This difference in strength means that the material cannot truly be considered as 'sandstone-like' as their paper suggests. It is possible that the increase in strength for the real material will mean that different failure mechanisms are available when compared with the lower strength material. If so, then the strength of such a model material must be significantly increased for it to simulate sandstone effectively.

In addition to the problem of creating artificial materials with the same, or sufficiently similar mechanical strength as real rocks, these techniques are limited by the difficulty of producing internal defects identical to those in natural rock. Structures cannot be exactly reproduced due to the impossibility of creating defects within the centre of a sample using moulds. However, the development of 3D printing has led

Fig. 4 A graph displaying how normalized breaking load varies with the number of cracks in samples made of a mixture of cement, sands, and water. Three samples were created for each number of cracks. The graph confirms that for a given number of cracks, the three samples exhibit a very similar breaking load, meaning that the relationship between the number of cracks and breaking load can be reliably obtained (Haeri et al. 2015)

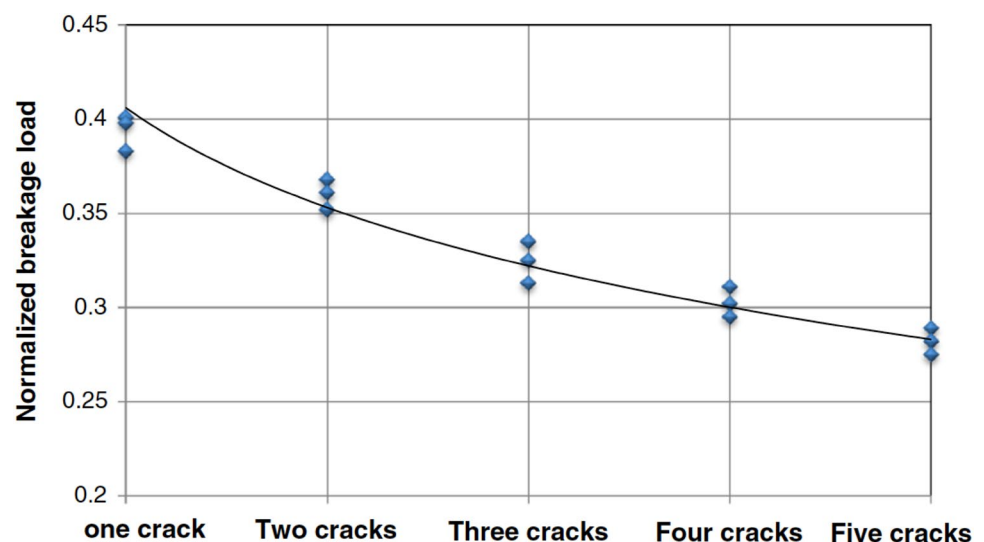
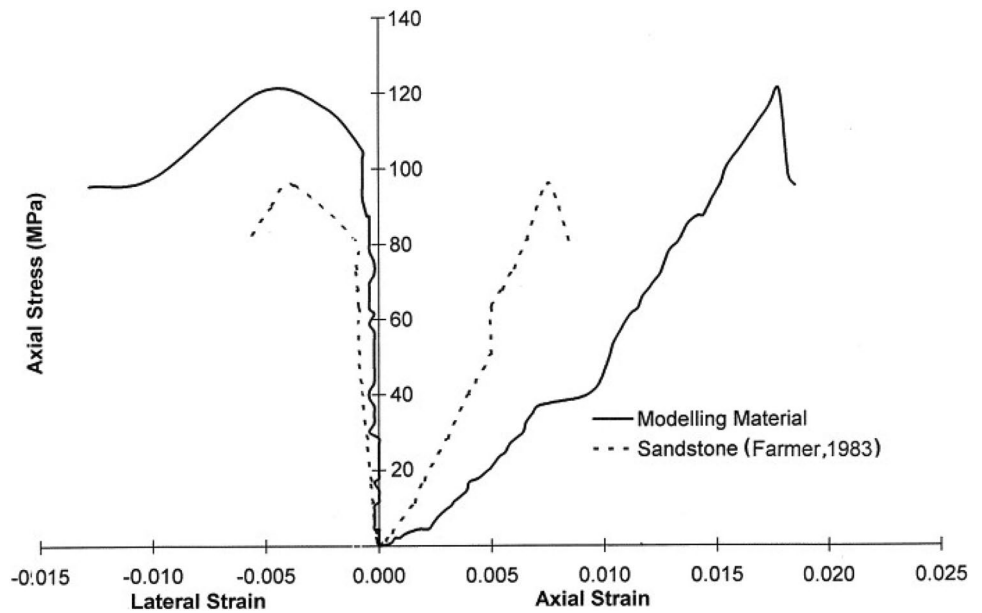


Fig. 5 A plot showing the stress–strain curves for sandstone (dashed line) and a modelling material comprised of barite, sands, plaster, and water (solid line). Both curves show a sharp fracture as strain increase, indicating brittle character (Wong and Chau 1998)



to recent attempts to overcome these problems using this technology (Bourke et al. 2008; Jiang and Zhao 2015; Tian and Han 2017a; Ji et al. 2018; Kong et al. 2018a; Sharafisafa et al. 2018; Squelch 2018; Zhou and Zhu 2018; Zhu et al. 2018a). The remainder of this article will therefore be a review of additive manufacturing to the investigation of rock mechanics.

4 Three-Dimensional Printing Technology

Three-dimensional printing technology (otherwise known as additive manufacturing) can be used to replicate the internal defect structure of rock masses (Suzuki et al. 2017; Kong et al. 2018b; Squelch 2018; Zhu et al. 2018a), something which is not possible using any previous technique for making artificial rock specimens. This technology makes

possible the creation of multiple identical samples so that tests can be repeated and hence more reliable conclusions drawn.

3D printing works by laying down layers of a chosen material, following the structure specified in a software file. In 2014, Ju et al. (2014) demonstrated the use of micro computed tomography (CT) to obtain the internal defect structure of coal samples, which they then implemented in the software of a 3D printer so as to print a number of identical polymer specimens containing the experimentally-measured defect structure. The images shown in Fig. 6 show the three stages in this process. The printed samples were made from two different polymers, one to represent the matrix and the other to represent the internal defects. This was a major improvement over homogeneous artificial rock simulants previously discussed. Ju et al. claimed that the overall mechanical properties of the polymers used in this investigation were ‘fairly close to that of natural coal rock’. The

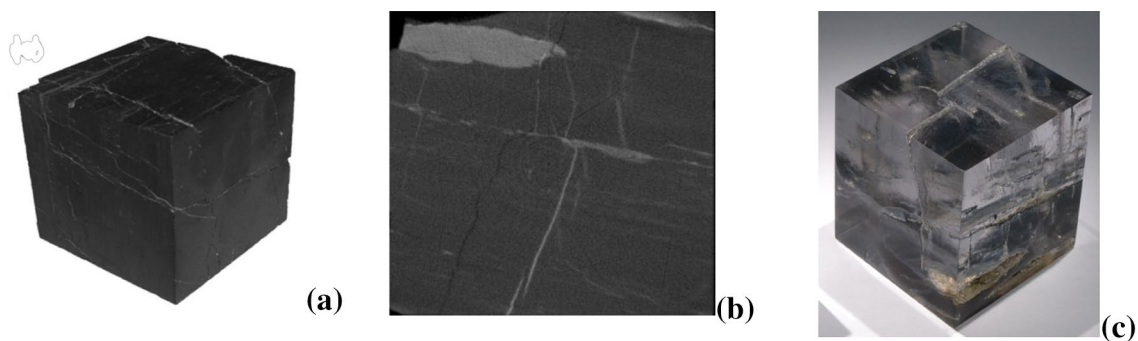


Fig. 6 Images showing how CT scanning is used to create identical printed specimens. **a** A real coal sample; **b** CT scan of **(a)**; **c** 3D printed polymer specimen created using information provided by the CT scan (Ju et al. 2014)

authors came to this conclusion due to the similarity of the values of the elastic modulus and Poisson's ratio of the coal and the 3D-printed polymer simulant. However, the UCS and tensile strengths of coal and the polymers they used are very different. Since the response to loading is affected by a wide range of mechanical properties, every property of the simulant that is relevant to the applied load needs to agree with the real rock of interest for their claim to be valid.

5 Studies of Materials So Far Used in 3D Printing of Rock Simulants

As just mentioned, the major problem with the polymers such as polylactic acid (PLA) that are currently used in 3D printing machines is their inability to adequately reproduce the mechanical properties of real rocks (Jiang and Zhao 2015). In particular the failure mechanisms that Jiang and Zhao saw in rock were not observed in 3D-printed PLA samples. Also the 3D-printed material had much lower strength and higher ductility compared to real rock samples. However, Jiang and Zhao were able to make their polymer specimens more brittle by printing samples with a lattice structure, in which the individual elements were connected in a web. This demonstrated the potential for amending the ductility character of materials through their internal structure and therefore may represent a way in which the field can develop in the future to achieve higher fidelity in terms of simulants. Another study showed that the mechanical properties of PLA are more similar to those of metals than rocks (Jiang et al. 2016b). The results suggest that to simulate rocks, 3D printable materials are needed that have a high compressive strength, low tensile strength, low ductility, and similar frictional properties.

More recently the validity of using a gypsum-like powder in this application has been investigated (Jiang et al. 2016a). Jiang et al. performed quasistatic tests to determine the basic mechanical properties of this rock simulant. The failure patterns were observed to be closer to natural rock than those made from PLA, as can be seen in Fig. 7. However, from this plot it can also be observed that the UCS of the gypsum-like material is much lower, indicating that a material needs to be developed to improve both properties simultaneously, and therefore, as with the sandstone mentioned earlier needs more development to improve this aspect of the material (or adequate validation to show that this is not important for a given loading regime of interest).

Jiang et al. also used high-speed photography to record the crack formation processes in printed samples. The photographic sequences they obtained showed crack coalescence and allowed the fracture pattern to be analyzed. The images show that some cracks coalesce from the tip of one preset crack to the middle of an adjacent one, and that cracks do not propagate from the tip of cracks that are perpendicular to the loading. The crack patterns were compared to those of a cemented sample studied by Haeri et al. (2014, 2015) and to those produced in numerical simulations (Fig. 8). While the patterns are quite similar, there are some differences regarding the location of the initiation of propagation. A further problem with these comparisons is that both the cemented sample and the numerical model were homogeneous materials, so the comparison does not confirm if the gypsum-based material displays the same crack fracture pattern as a real heterogeneous rock. Comparisons with more homogeneous rock specimens might be more favourable, but such validation still needs to be performed.

Comparisons have begun to be made (Fig. 9) between the propagation of cracks in 3D-printed and real rocks so as to

Fig. 7 A comparison of the stress–strain curve of a 3D-printed PLA sample (dashed line) with the gypsum-like 3D-printed sample (solid line) (Jiang et al. 2016a)

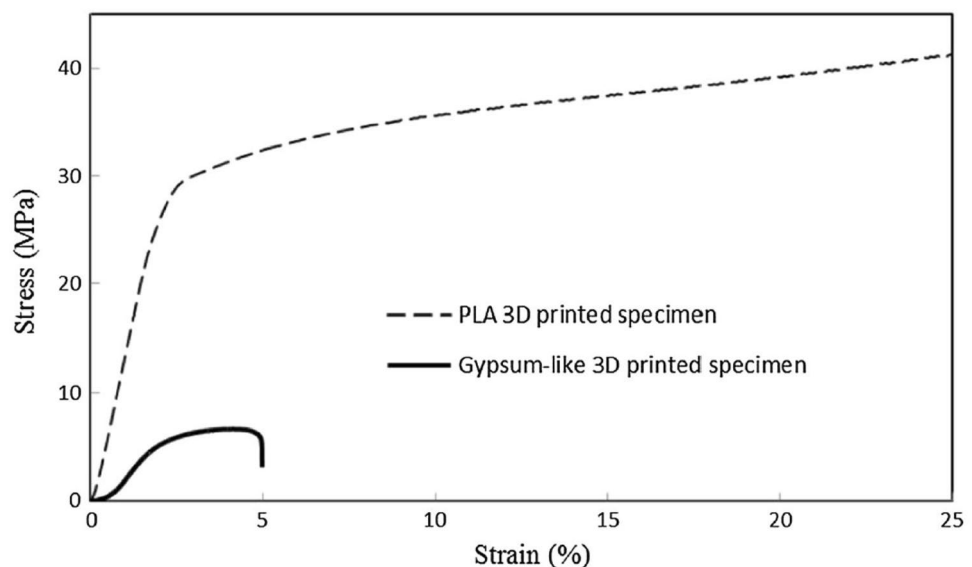
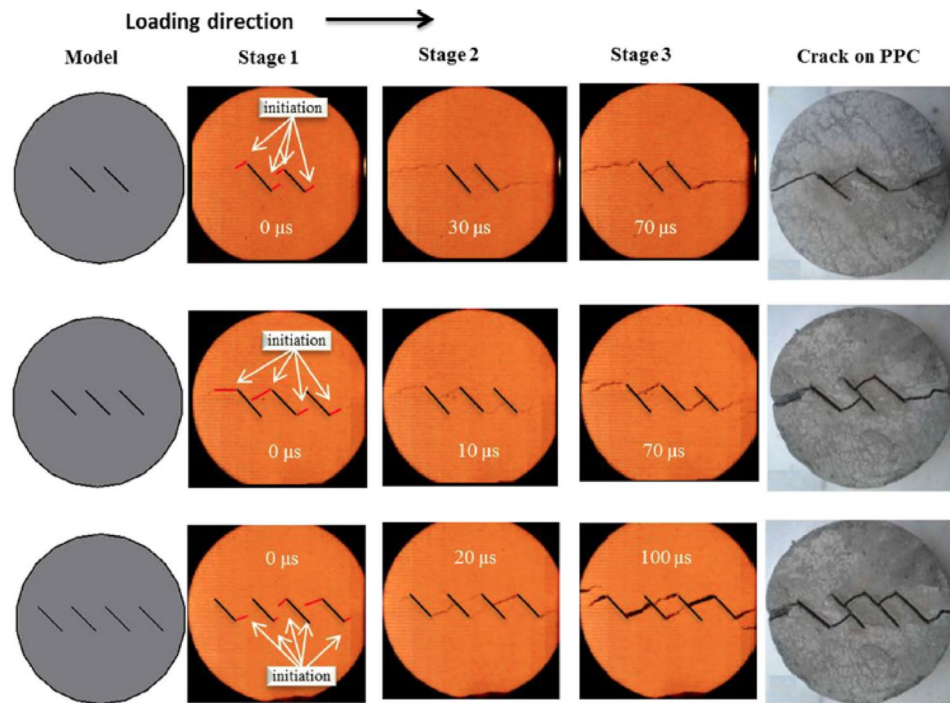


Fig. 8 Images in orange show the coalescence of different arrangements of cracks in three different models compared to that of the same cracks created in a Portland pozzola cement (PPC) sample (Jiang et al. 2016a). Adapted from Haeri et al. (2014)



determine whether cracks coalesce in the same way (Tian and Han 2017a, b). Squelch (2018) has recently said that “3D printing technology has reached a sufficiently mature stage that it is possible to print 3D models of rocks and rock surfaces with adequate geometrical and visual realism”. However, he goes on to say “The level of realism of 3D printed models of rock does not extend to the full geological character and physical property of the original rock material”. But in a paper published about the same time by Tian et al. the stress–strain response of rock simulant specimens 3D-printed using sand can be seen to be very similar to real sandstone (Fig. 10). Their results give hope that this relatively novel technique of making artificial rock-like specimens has the potential of allowing specimens to be made with mechanical properties much closer to those found in nature than previous techniques, particularly for materials which are naturally relatively homogeneous aggregates of bonded grains.

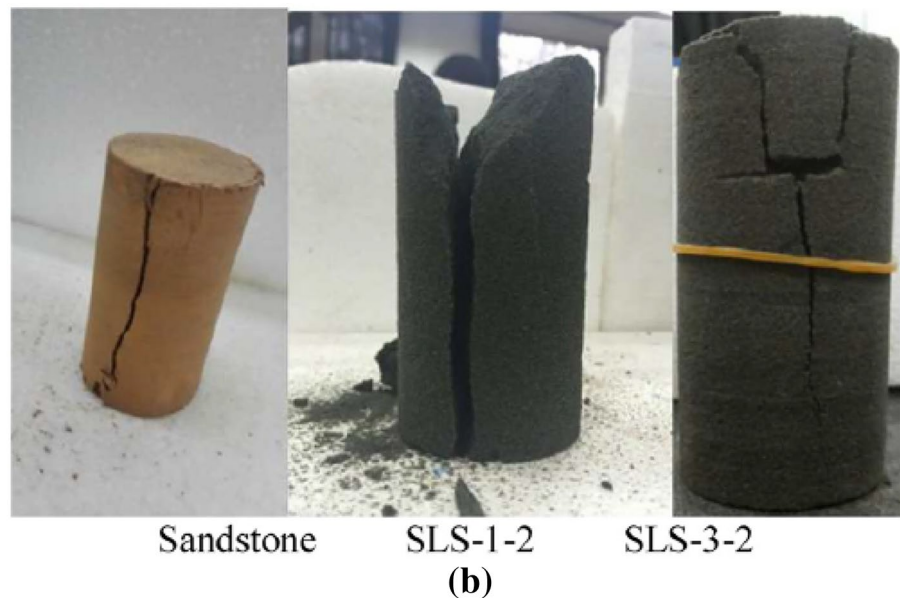
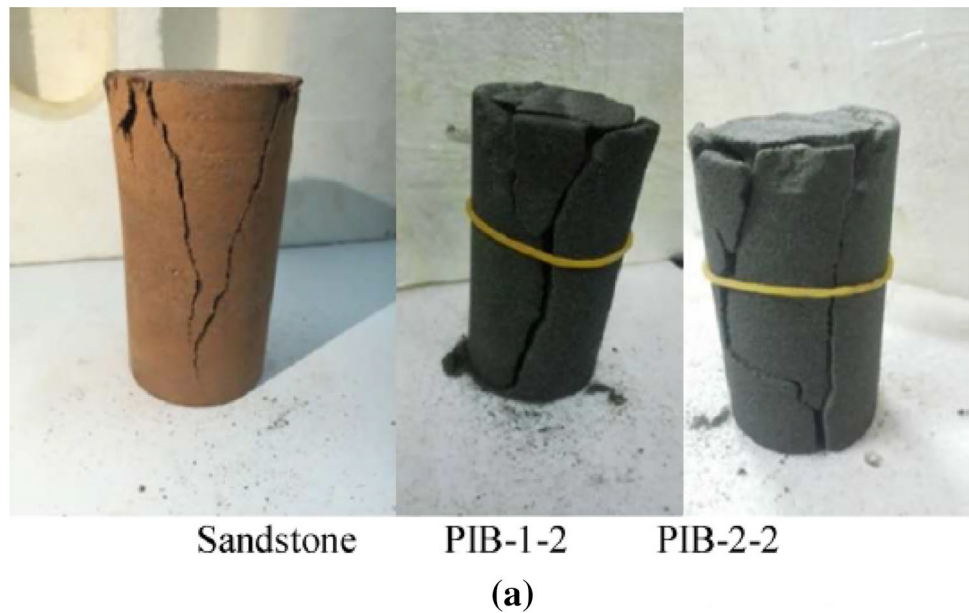
Since ceramics can now be 3D-printed (Goulas et al. 2016; Costa et al. 2017; Goodman 2017; Singh et al. 2017; Ferrage et al. 2018; Owen et al. 2018) they should be considered as rock simulants since they have various mechanical properties that are closer to rocks than polymers. Up to the time of writing there have been no published investigations of the use of printed ceramic or natural rock materials in rock mechanics studies (Zhou and Zhu 2018). One problem may be that the thermal gradients produced using a laser sintering ceramic printing process often create unwanted cracks. However, it has been shown that it is possible to create ceramic samples by the pyrolysis of printed liquid

resin samples, resulting in specimens with a compressive strength of 163 MPa (Eckel et al. 2016). This suggests there may be great potential for the creation of high strength rock simulants by low temperature 3D printing, which should therefore definitely be investigated for use in rock mechanics studies.

6 Effects of Printing Techniques and Post-processing

The properties of 3D-printed samples have been shown to be affected both by the printing technique used and subsequent processing (Vorndran et al. 2015). This opens up the possibility of altering the characteristics of printed specimens to make them more accurate simulants of real rocks. In one investigation four specific topics were investigated: (i) anisotropy due to layer printing, (ii) binder saturation level, (iii) printing layer thickness, and (iv) heat treatment (Fereshtenejad and Song 2016). This study showed that cylindrical samples with vertical layers (which they described as having an inclined angle of 0°), increased binder saturation and a heat treatment of 60 min all produced higher ultimate compressive strengths (UCS) and more brittle behaviour (Figs. 11, 12 and 13). Figure 12 also shows that the layer thickness had no clear effect on strength. However, Fig. 12 does show that constant binder saturation specimens with thicker layers were more likely to have a higher strength. The highest UCS was observed for specimens that had been heated to 150°C (Fig. 14), but the most brittle behaviour was seen at 70°C

Fig. 9 Comparison of failure of rock and 3D-printed rock simulants (Tian and Han 2017b)



(Fig. 15), suggesting that an optimal temperature needs to be chosen to balance the two effects. One conclusion of this research is that heat treatment is not an effective method for modifying 3D-printed polymer-bonded ceramic materials to simulate rocks as both a high UCS and a high brittleness are required. So further research must be performed to find a way to achieve desirable values of both these properties.

In another investigation the properties of transparent resin-printed samples were improved during the preparation process (Zhou and Zhu 2016). The samples were frozen to $-73.5\text{ }^{\circ}\text{C}$ and printed with an internal square crack within. These samples then showed high brittleness and an increased UCS of 81.3 MPa, which is similar to rocks. In the testing process, the specimens still displayed elastic deformation

at the beginning of the loading process which meant cracks developed more slowly than in the rock samples studied (Zhou and Zhu 2017). Whilst this shows that the printed samples were still not identical to rock, the improvement obtained in the brittleness and strength shows there is potential for developing the technique further.

7 Print Resolution

A final important factor when investigating the usefulness of a given 3D printing technique is the minimum resolution at which the samples can be printed, as this determines whether the rock microstructure can be recreated down to

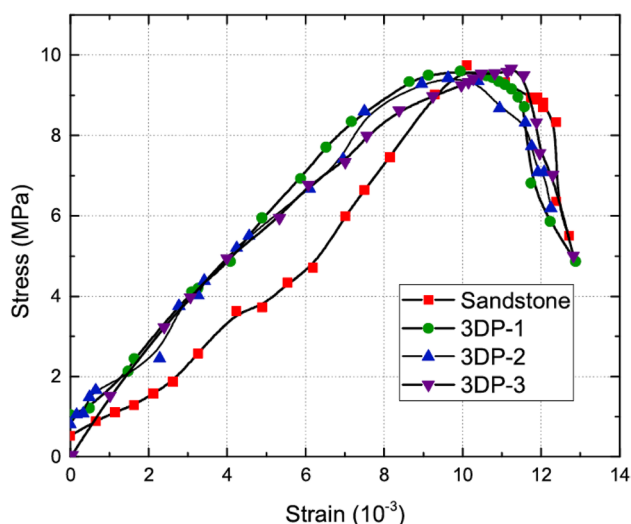


Fig. 10 Comparison of stress–strain curves for a sandstone with specimens 3D-printed using sand and a resin (Tian and Han 2017b)

the scale of the grains. The resolution must be considered in all three dimensions, the x and y axes being in the plane of the layers and the z axis being normal to the layers. The z axis resolution is determined by the thickness of the layers, whereas the x – y resolution is more complicated and can vary between different printers. Different prototyping techniques exist with differing x – y resolutions (Rengier et al. 2010). For example, stereolithography, where photopolymers are made using UV lasers, results in a much higher x – y resolution than inkjet printing, which is commonly used for layering fine powders. The technique that is used in any given application largely depends on the material being printed.

The resolution in the x – y plane is clearly therefore just as important as the layer thickness, but many of the papers

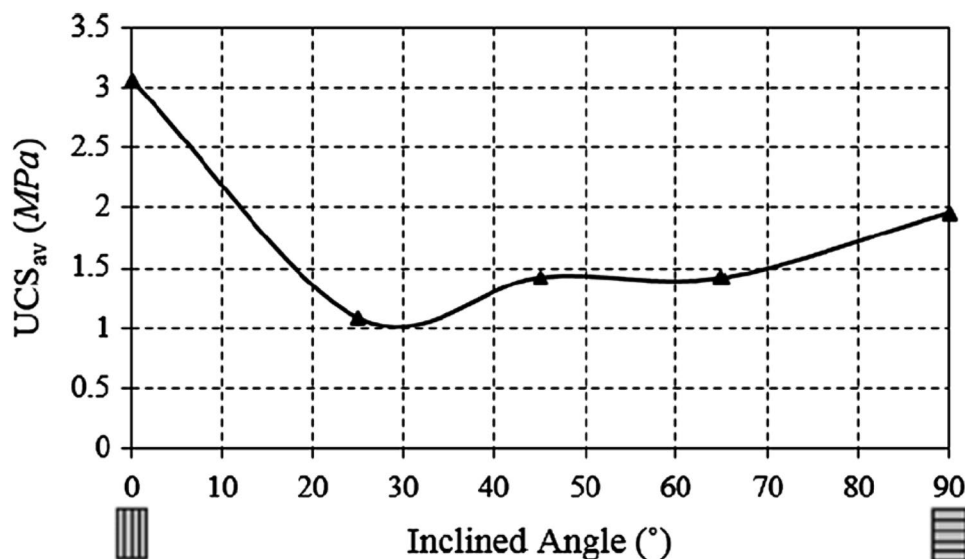
reviewed include only the layer thickness and no information about the prototyping technique used. A layer thickness of 0.1 mm appears to be the most commonly quoted value (Jiang et al. 2016a, b), although higher resolution stereolithography printing mechanisms with a layer thickness of 50 μm have been investigated (Zhou and Zhu 2016, 2017). The Wentworth classification system for the grain sizing of sands (Wentworth 1922) defines fine sand as having a grain size between 0.063 and 0.2 mm. So layer thicknesses within this range should be able to contain most grains of sand. 3D printing could therefore be used to recreate defects within and the fabric of rocks whose grains are composed of sand or coarser materials, but it cannot be used to accurately recreate rocks containing grains derived from silt. However, the x – y resolution also needs to be specified to confirm this conclusion is valid for all dimensions. The resolution in all three dimensions should be quoted in future publications to confirm the validity of the results reported.

8 Conclusions

Overall, it is evident there are advantages in using both artificially moulded and 3D-printed specimens in place of natural rock in mechanical tests. Both methods allow the production of samples for testing that contain specific features such as cracks. 3D printed samples have the further benefit that the internal defect structures of real rocks can be repeatably reproduced down to the level of individual grains. This cannot be achieved using moulding processes.

The crucial problem with the studies so far published is that the artificial samples produced do not, even in many cases by the authors' own admission, satisfactorily simulate the mechanical properties of rock. In recent years, however,

Fig. 11 Plot showing the effect of printing direction on the ultimate compressive strength (UCS) of 3D-printed specimens (Fereshtenejad and Song 2016)



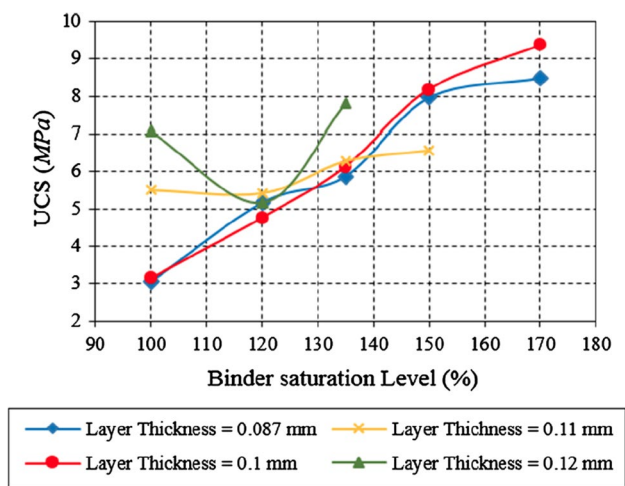


Fig. 12 UCS of 3D-printed specimens having various layer thicknesses and binder saturation levels (Fereshtenejad and Song 2016)

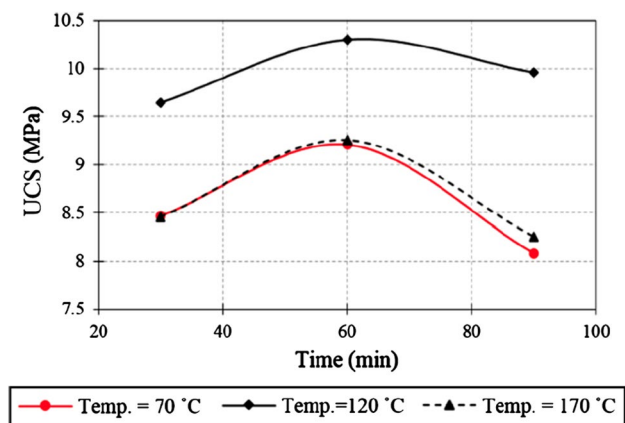


Fig. 13 Plot showing the effect of heating duration on the strength of 3D-printed specimens (Fereshtenejad and Song 2016)

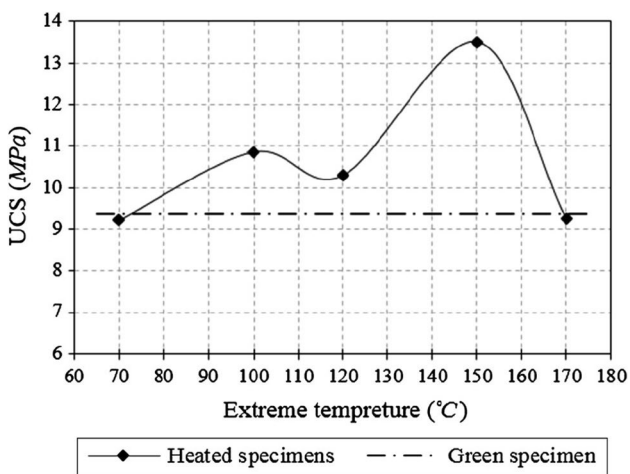


Fig. 14 UCS of 3D-printed specimens after heating for 60 min at various temperatures (Fereshtenejad and Song 2016)

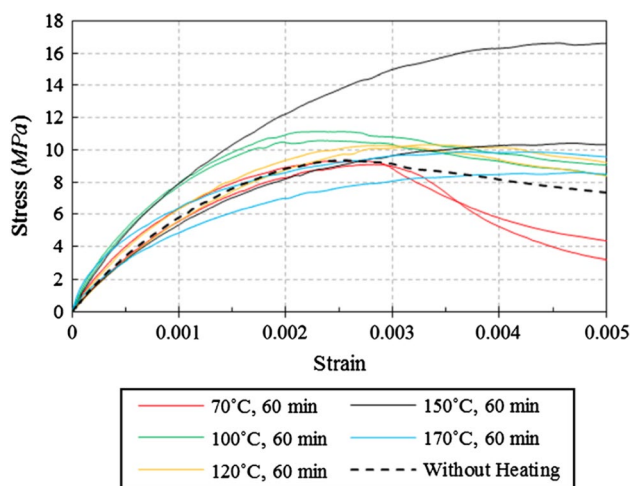


Fig. 15 Stress–strain graphs of UCS tests conducted on heated 3D-printed specimens (Fereshtenejad and Song 2016)

investigations have shown the need for simulant materials with high strength and low ductility. It has also been shown that printing and post-processing techniques have the potential to improve these properties, through processes such as lowering the temperature and changing the angle that successive layers are printed at. Whilst it is unrealistic to expect artificial specimens to perfectly simulate real rocks, the significant recent improvements suggest there is the potential to produce simulant materials with much more similar mechanical properties to rocks than previously. Specimens made of such materials would enable more repeatable (and hence statistically more accurate) tests to be performed which would provide better insights into the mechanical properties of rocks, particularly their fracture properties.

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