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Linking micro-mechanical measurements to macro-mechanical properties.

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Summary

This paper presents a study of the effectiveness of the plasticity effect model, and the cumulative stress transfer function (CSTF) approach to data reduction in the fragmentation test, as a technique for determining the interphase characteristics of two types of carbon fibre. The experimentally determined mechanical properties of high volume-fraction composites have been compared with predictions from single filament tests analysed using these techniques. It is found that the explicit consideration of elasto-plastic deformation of the interphase and debonding, which are incorporated into the plasticity effect model, allows the results of single-fibre and high volume-fraction composites to be correlated in certain testing geometries. In cases where correlation is impossible using only the CSTF value, analyses based on finite element modeling of the fragmentation test can be applied in order to infer the interphase properties. Once the properties of the interphase are established, prediction of the performance of high volume-fraction composites tested in other geometries is shown to be possible.

Keywords: Composite properties, Fragmentation testing, Interphase, Plastic deformation.

Introduction

For many years single-fibre model composites have been used to characterise fibre/matrix adhesion. In theory, these data should correlate with the performance of high volume-fraction composites produced using the same fibres and resin, particularly in the longitudinal direction. However, up to now the data reduction techniques that have been used to analyse single-fibre composites have not been sufficiently accurate for any direct links to be established. The fragmentation test is the most popular single-fibre test geometry as it involves loading the fibre analogously to that experienced in longitudinal high volume-fraction composite materials. The data reduction techniques used in the analysis of fragmentation data have traditionally been the Kelly-Tyson approach[1], first proposed in 1965 for the analysis of metal-matrix composites, and the earlier Cox model[2]. Neither of these techniques fully accounts for the observed strain-transfer processes. The Kelly-Tyson approach[1] assumes the strain transfer to be fully plastic in nature, whereas the Cox model[2] assumes elastic strain transfer. More recently other approaches have been developed which aimed to overcome difficulties in these two classic analyses, particularly by incorporating debonding[3,4]. However, all of these fail to overcome the fundamental problems associated with the assumptions of perfectly plastic or perfectly elastic deformation, because real systems display a mixture of the two. Tripathi et al.[5] developed a model, termed the plasticity effect model, in which the assumptions of either fully elastic or fully plastic deformation were overcome by explicitly incorporating the elasto-plastic nature of the matrix and the occurrence of debonding. In order for the plasticity effect model to act as a data reduction technique and to determine the interphase quality, the ability of the fragments to sustain load was assessed. From this a numerical value was generated which is termed the cumulative stress transfer function (CSTF)[6]. As the CSTF value accounts for elasto-plasticity and debonding, it should overcome the limitations of the previous analyses.

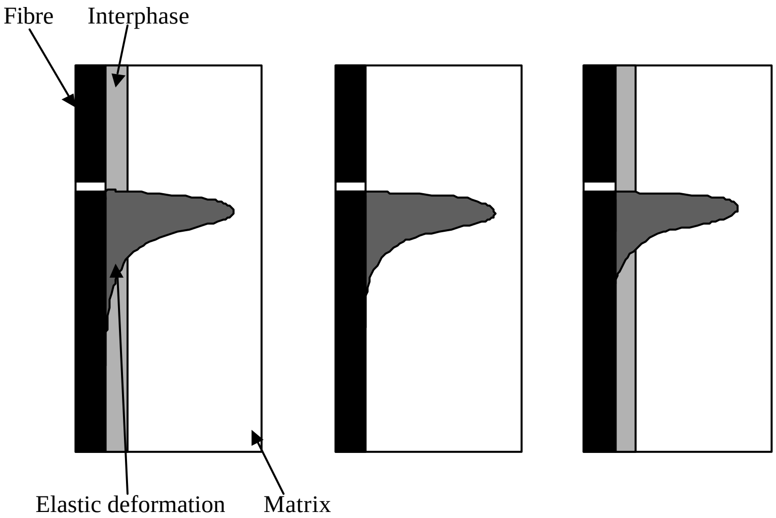
A number of authors have attempted to link results from micro-mechanical testing to results obtained from high volume-fraction composite[7,8,9], with limited success. Drzal and Madhukar[7] carried out an analysis using three types of carbon fibres, which differed only in their surface treatment. These were tested as single-fibre model composites and also high volume-fraction composites in a variety of geometries. It was found that the fibre/matrix adhesion, which was determined at saturation of the fragmentation process, combined with an analysis of the observed failure-modes, enabled interpretation of the results obtained from high volume-fraction systems. However, it must be stressed that the interpretation of the results relied not-only on the fibre/matrix adhesion data as deduced from the Kelly-Tyson approach, but also on the observed failure processes. Ivens et al.[8] studied the effect of surface-treatment level on the adhesion of unsized carbon-fibres by comparing single-fibre data with data from longitudinal tensile testing. It was found that the maximum adhesion as measured from single-fibre composites was not necessarily desirable in order to obtain the maximum modulus or strength in the high volume-fraction composite, as brittle failure could be induced. Hoecker and Karger-Kocsis[9] carried out both micro-bond and fragmentation testing to measure fibre/matrix adhesion. They compared the fibre/matrix adhesion characteristics with the transverse properties, shear properties and impact performance of high volume-fraction composites. Their findings showed that the micro-bond test was more easily related to the nature of the interphase than the fragmentation test. They also found that the level of adhesion between fibre and matrix could be related to the failure strengths in these geometries, particularly in relation to whether failure occurred interfacially or cohesively in the matrix. Thus, it has proven possible to link data from the fragmentation test to selected measurements on high volume-fraction composites, but these relationships are purely qualitative and rely largely on observation of the failure processes, not solely on the adhesion parameter. A method based purely on a data reduction technique is preferable, as less emphasis is placed on the skill of the operator and automation of the analysis becomes practical.

This paper aims to fully investigate the CSTF approach to interphase characterization and to examine whether it is sufficiently accurate to allow relationships between single-fibre and high volume-fraction systems to be established using purely the CSTF value. To achieve this, a study using two fibre types, one sized the other unsized is undertaken using both single-fibre systems and high-volume-fraction composites. The analysis employs finite element modeling of the effect of an interphase on stress transfer characteristics which have been discussed in detail elsewhere[10,11].

The prior art.

The authors have previously conducted research in to the effects of an interphase in both single-fibre[10] and high volume-fraction[11] composites. The studies have been based around finite element analysis, although the results in the case of single-fibre systems have been experimentally verified.

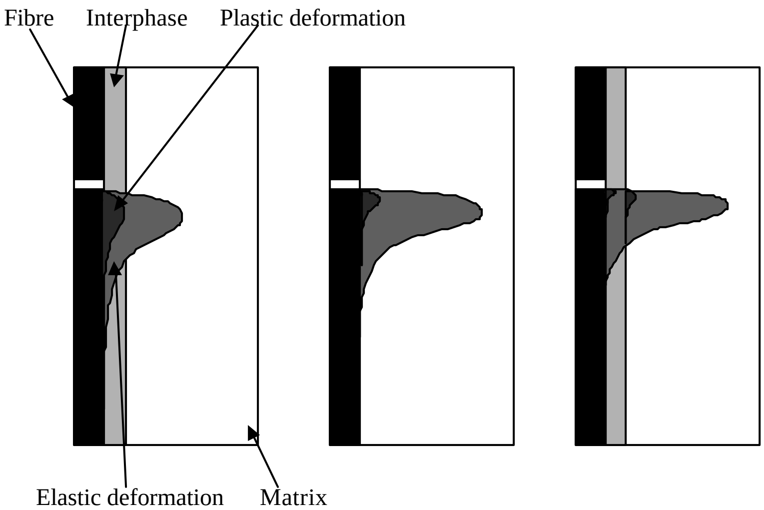
Considering single-fibre model composites with a thin discrete interphase of known properties, at low applied strains when deformation is elastic the mechanical properties of the interphase have a relatively insignificant effect on the level of strain transfer to the reinforcing fibre. Therefore, in these circumstances, the matrix dominates the strain development within the fibre, and the deformed zone around a fibre-break is the same size whether the interphase is soft, stiff or non-existent (Figure 1). However, at higher applied strains when plastic deformation starts to occur the interphase properties become significant and the size of the deformed region depends on its properties (Figure 2). In the presence of a soft interphase (Figure 2a), yield will occur in the interphase and the rate of strain transfer to the fibre will be substantially reduced, the size of the deformed region in the matrix will also decrease, as energy is absorbed in the interphase. Conversely, in the presence of a stiff interphase (Figure 2c), yield will be less likely to occur within the interphase itself, leaving the deformed region largely unaffected, and the strain transfer to the fibre will be similar to that observed in the absence of an interphase (Figure 2b). As the interphase thickness increased, its influence on



the mechanical properties increased, due to its increasing volume-fraction.

a b c

Figure 1. Schematic illustration of the deformation within the matrix and interphase at low applied strain, when elastic deformation dominates, in the presence of a) a soft interphase, b) no interphase and c) a stiff interphase.



a b c

Figure 2. Schematic illustration of the deformation within the matrix and interphase at high applied strain, when elasto-plastic behaviour becomes significant, in the presence of a) a soft interphase, b) no interphase and c) a stiff interphase.

Having established the effect of plastic deformation within an interphase in single-fibre systems, a 3-dimensional finite element analysis examined the significance of plasticity in high volume-fraction systems. The increased constraint imposed by the surrounding fibres was found to promote plastic deformation even at the low strains experienced by most engineering composites. The effect of the interphase on the level of strain concentration in neighbouring fibres can be seen in Table 1, where the strain concentration factors for various systems are presented. The influence of the interphase is most apparent when it is soft, as yield within the interphase reduces the strain concentration experienced by fibres surrounding a broken one. This reduction in the strain concentration factor will reduce the likelihood that a cluster of fibre-breaks will occur and, thus, failure of the composite will be delayed. The effect of a stiff interphase in increasing the strain concentration is not as great, because the matrix resin then absorbs more energy and reduces the strain concentration to the level that would be expected if the interphase were not present. This work was purely based upon 3-dimensional finite element analysis.

Table 1. Strain concentration factors for various matrix and interphase mechanical property combinations, at a volume-fraction of 58%.

	Matrix/interphase properties			
Deformation process	**Stiff/Not present**	**Stiff/Soft**	**Soft/Stiff**	**Soft/Not present**
Elastic	1.15	1.13	1.13	1.12
Plastic	1.07	1.05	1.05	1.05

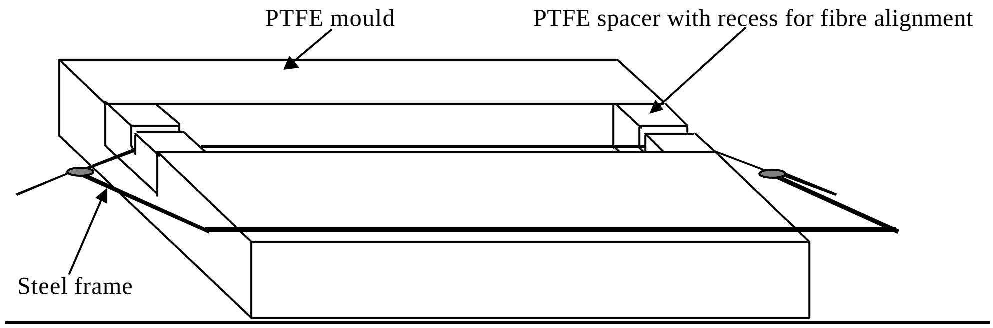
Experimental

Materials.

The experimental study of both high volume-fraction and single-fibre model composites required the use of two carbon-fibres one with sizing and one without, but which were otherwise identical. To this end, Tenax HTA 5001 and HTA 5131 were employed, these being type 'A' carbon-fibres which have received an electrochemical oxidative treatment in both cases and a sizing resin in the case of HTA 5131. This ensured that the strength and moduli of the two fibres had a negligible influence in this study[10]. A single matrix system was selected for this study, a medium temperature curing resin from the Advanced Composites Group Ltd designated MTM 60. In this way, the differences in the two composite samples examined in this study were limited to the effect of the sizing resin, allowing its influence to be fully investigated.

Fragmentation testing

Single fibres were separated from the tow and mounted across a metal frame, facilitating handling of the fibre and also ensured its alignment during embedding within the resin. Liquid resin was heated to reduce it viscosity and degassed in a vacuum oven. PTFE moulds with a rectangular cavity were employed, the hot resin being poured into the cavity prior to inclusion of the fibre. Fibres were embedded into the centre of the moulding by placing them, still attached to the frame along the central portion of the mould, where recesses in the mould had been located, the weight of the frame acting to align the fibre and to pull it into the centre of the mould cavity (Figure 3). The samples were then cured using the manufacturers recommended cure schedule of 30 minutes at 60°C followed by ramping to 120°C at 20°C per hour and holding at 120°C for 90 minutes. Samples were then allowed to cool naturally to room temperature. Once cured, the samples were carefully polished to a uniform section of 80 mm by 9.8 mm by 1.65 mm, ensuring a good optical surface for observation in the



fragmentation test.

Figure 3. Schematic illustration of the mould used to produce fragmentation specimens and the manner in which the mounting frame was used to aid in fibre alignment.

Fragmentation testing was carried-out using a custom-built miniature tensile-testing frame, incorporating a video-capture microscope. This allowed pictures of the fragments to be rapidly captured and stored for subsequent analysis. A testing speed of 0.13 mm/minute was employed, the samples being progressively strained in 1% intervals. After each interval, the testing was paused and the sample examined to see if fragments had formed. Any fragments were photographed, or measured using a video-micrometer if they exceeded the field width. The photographs were analysed to measure the fragment and debond lengths using custom-written software, the results being output as a text file. Using further software, these files were processed to calculate the CSTF value, allowing rapid analysis of fragmentation data.

High volume-fraction composites

High volume-fraction composites were produced using a laboratory drum-winder. ACG provided MTM 60 in the form of resin-film with an area density of 50 g/m^2. A metre of resin film was wound around the drum of the winder and the drum-heater was set at 40°C to soften the film. Fibres were wound on to the tacky film, the rate of drum rotation and fibre traverse being controlled to give a final volume-fraction of approximately 60%. A second resin-film was then wound onto the fibres, and the whole structure was heated to 40°C in a vacuum-bag. This process facilitated a gentle infusion of the resin into the fibres, maximising the consolidation of the pre-preg prior to lay-up and cure of the composite in an autoclave. Analysis revealed that composite produced in this manner was comparable to commercially available pre-preg produced using the same type of resin and fibres[12].

Samples were produced using composites manufactured from both fibre types and tested in the following geometries:

Longitudinal tensile testing (BS2782 (1976))
Transverse tensile testing (ASTM D3039 (1976))
Three point flexural testing (EN ISO 14125 (1998))
Short beam shear testing (ASTM D2344 (1984))

All testing was carried out in an air-conditioned room set to 20°C and 50% relative humidity. Full analysis of the results were carried out to allow comparison of the effect of the interphase and also to enable cross-comparison with the fragmentation test results.

Results

Fragmentation testing

A summary of the fragmentation test data is shown in Table 2, full details of the measurements can be found in reference 12. It should be noted that the HTA 5001 fibres displayed significant debonding while HTA 5131 did not.

Table 2. Summary of results from fragmentation testing.

	HTA 5001 (unsized)					HTA 5131 (sized)				
Applied Strain (%)	4	5	6	7	8	4	5	6	7	8
Avg. fragment length (mm)	0.91	0.38	0.34	0.33	0.32	0.68	0.34	0.27	0.24	0.23
Avg. debond length (mm)	0.004	0.02	0.03	0.04	0.07	--	--	--	--	--
CSTF value (MPa)	3863	2410	1796	1544	1374	2974	2254	1871	1629	1533

In order to compare the quality of the adhesion using the CSTF approach, the values for the two sample types are plotted in Figure 4 against the applied strain.

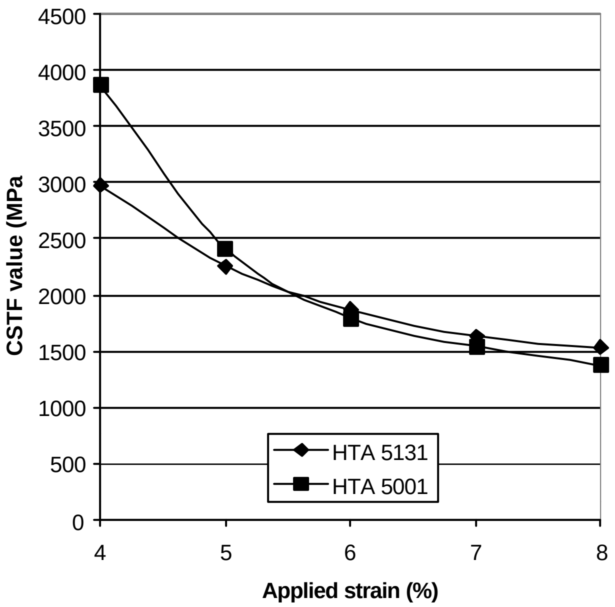


Figure 4. Graph showing the trends in CSTF value with increasing applied strains for the two systems.

In Figure 4, it can be seen that initially the CSTF value of the HTA 5001 fibres exceeds that of the HTA 5131 fibres. However, at higher strains, the CSTF value of the HTA 5001 falls below that of the HTA 5131. The nature of the CSTF value dictates that both curves reduce in value as the fragment length decreases, reducing the fibres ability to sustain load. Generally, the CSTF value can be considered to indicate the strain carrying ability of the fibres and, thus, a high value indicates better reinforcement. The effect of plasticity is to reduce the load carrying capacity of the fibre and therefore reduce the CSTF value. The presence of debonding also reduces the load transfer between matrix and fibre and, therefore, reduces the CSTF value, in fact reducing it more than the occurrence of plastic deformation. Therefore, the plot shown in Figure 4 provides a detailed description of the respective failures within the two systems. As it considers plasticity, which is inherently a bulk effect, the CSTF value is not indicative only of the interface strength, but must be considered as a system parameter indicating the strain transfer performance in a global manner. Therefore, differences in the interphase performance can be readily extracted from the results using this technique, as the only variable is the interphase.

Using the CSTF approach and previous analyses[10,11], the following interpretation can be developed. Initially the HTA 5001 fibres display a higher CSTF value than the HTA 5131, suggesting that it has a rigid interphase. However, as the strain is increased, failure of this stiff, but relatively brittle, interface occurs leading to debonding in the case of HTA 5001. The softer interphase of the HTA 5131 fibres does not debond and therefore its ability to transfer strain is unimpaired, meaning that its CSTF value is higher at the end of the test than that of HTA 5001. Therefore, we can establish that the effect of the sizing resin in the HTA 5131 based composite is to provide a soft, resilient, interphase.

Prediction of the properties of high volume-fraction composites

Having gained a knowledge of the interphase properties, it is possible to develop a series of predictions relating the interphase to the properties of high volume-fraction composite systems. However, the fibre matrix adhesion level, as summarized in the CSTF value, can only be directly related to the longitudinal strength of the composite, as it is derived from this orientation. To predict the properties of a composite tested in other orientations, consideration of the inferred interphase properties is necessary.

Property predictions made using the CSTF value

The CSTF curve plotted in Figure 4 can be directly related to the strength of a high volume-fraction composite produced using these fibres and tested in the longitudinal direction. The strength of the composite depends on the strain transfer processes at higher applied strains, even though the failure strain of the composite is substantially below those experienced in the fragmentation test. This is because the constraint present in high volume-fraction systems leads to an effective strain magnification and therefore the occurrence of debonding and plasticity at lower strains is observed in high volume-fraction composites. From Figure 4 it can be seen that the strain transfer capability of the HTA 5131 decreases less than that for the HTA 5001 as strain increases and therefore a higher strength is predicted.

Property predictions made with reference to the inferred interphase properties

A summary of the predictions for all geometries except the longitudinal strength, along with the rationale used in making them, is included in Table 3. The predictions are made with reference to the inferred properties of the interphase which were discussed above, as this will dictate the deformation and failure processes which occur.

Table 3. Summary of the predicted effect of a soft interphase on selected mechanical properties.

Property	**Prediction**	**Rationale**
Longitudinal modulus	HTA 5001 > HTA 5131	Soft interphase in HTA 5131
Transverse strength	HTA 5131 > HTA 5001	Low interface adhesion in HTA 5001
Transverse modulus	HTA 5001 > HTA 5131	Soft interphase in HTA 5131
Flexural strength	HTA 5001 > HTA 5131	Soft interphase in HTA 5131
Flexural modulus	HTA 5001 > HTA 5131	Soft interphase in HTA 5131
ILSS	HTA 5131 > HTA 5001	Low interface adhesion in HTA 5001

High volume-fraction systems

Table 4 shows a summary of the measured mechanical properties of the two composite systems, along with the standard deviation in the measurements.

Table 4. Summary of the mechanical properties of the two composite systems (standard deviation in brackets).

Property	**HTA 5131**	**HTA 5001**
Volume-fraction (%)	67.2	64.0
Longitudinal strength (MPa)	1614 (80)	1497 (48)

Longitudinal modulus (GPa)	163 (9)	158 (13)
Transverse strength (MPa)	53 (4)	39 (6)
Transverse modulus (GPa)	9.5 (1)	9.3 (1)
Flexural strength (MPa)	1686 (138)	1807 (97)
Flexural modulus (GPa)	122 (8)	120 (9)
ILSS (MPa)	66 (3)	63 (3)

It can be seen that the volume-fractions of the two composites differ significantly and hence it is difficult to directly compare the differences in their longitudinal properties. Therefore the longitudinal properties were scaled to a constant volume fraction of 64.6 %, the results being shown in Table 5.

Table 5. Summary of the scaled longitudinal properties of the two composite systems (Standard deviations in brackets).

Property	HTA 5131	HTA 5001
Longitudinal strength (MPa)	1551 (77)	1510 (48)
Longitudinal modulus (GPa)	157 (9)	159 (13)

Table 5 shows that the difference between the longitudinal strengths of the two systems is small, with the HTA 5131 displaying a slightly higher mean value. However, the difference in the CSTF values for the two systems are also small (Figure 4) and so this small improvement in favour of the HTA 5131 is to be expected and the prediction made previously is qualitatively correct. From Tables 4 and 5, it can be seen that the other predicted relationships between the HTA 5131 and the HTA 5001, are also realised. It is therefore apparent that the use of the fragmentation test to analyse failure in high volume-fraction composites is indeed possible. However, careful data reduction is necessary in order to ensure that all of the failure mechanisms, such as plasticity in the interphase, or matrix, and also the occurrence of debonding, are accounted for. This study shows that the plasticity effect model and the CSTF analysis represent a significant improvement over conventional analyses for the fragmentation test, as they explicitly consider debonding and plastic deformation. By using the CSTF value it was possible to observe the progressive failure of the interphase under increasing applied strains and therefore to understand the breakdown of high volume-fraction composites under longitudinal load. As a result of this it was possible to interpret the fragmentation test in such a way that the characteristics of the two systems considered in this study could be accurately distinguished. Also, by using the CSTF analysis combined with findings from previous studies[10,11], it was possible to interpret the results in such a way that the properties of the interphase could be determined qualitatively. This enabled a more detailed understanding of the composites properties in geometries other than the longitudinal than would otherwise have been possible. Once the in-situ properties of the interphase can be quantitatively determined the development of more detailed models for predicting composite properties will be possible. Work is continuing in this direction.

It is worthy of note that the increase in longitudinal strength represents a 2.6% increase, while the modulus reduction is less than 1%. Similar findings are made in the analysis of the transverse properties. Therefore, it appears that the application of a sizing to the fibres provides an effective means to influence the toughness of a composite, while minimizing the detrimental effects associated with the use of a softer matrix.

Conclusions

Using the CSTF approach to analyse single-fibre fragmentation tests it has been possible to directly relate the interphase quality to the longitudinal properties of high volume-fraction composites. Explicit consideration of the plastic deformation in the matrix, and also the presence of debonding, has allowed a more thorough understanding of the performance of a sizing resin than would previously have been possible. As a result of the CSTF analysis it was possible to predict the effect of a sizing resin on the longitudinal strength of the composite. The prediction was found to be qualitatively accurate, although quantitative predictions were not attempted.

Using the CSTF approach and also the results of finite element analyses conducted by the authors it has been possible to not only obtain an indication of the effect of the interphase on the longitudinal strength of the composite, but also to make predictions regarding the mechanical properties of the interphase region itself. Once an understanding of the interphase properties had been obtained, it was then possible to predict its effect on the composite properties in other testing geometries. Again, the predictions were found to be qualitatively correct. The determination of interphase properties in this way is a significant advance in the development of models to quantitatively predict the properties of high volume-fraction composites.

Acknowledgments

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