Role of nano-silica in tensile fatigue, fracture toughness and low-velocity impact behaviour of acid-treated pineapple fibre/stainless steel wire mesh-reinforced epoxy hybrid composite

T Dinesh 1, A Kadirvel 2 and P Hariharan 3

1 Department of Mechanical Engineering, J. N. N. Institute of Engineering, Anna University, Chennai-601102, India
2 Department of Mechanical Engineering, R. M. K. Engineering College, Anna University, Chennai-601206, India
3 Department of Manufacturing Engineering, College of Engineering Guindy, Anna University, Chennai-600025, India

E-mail: dinesht@jnn.edu.in
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Abstract
Nano-silica toughened epoxy hybrid composites were prepared along with pineapple fibre and stainless steel wire-mesh and characterized for its tensile fatigue, fracture toughness and low-velocity impact behaviour. The principal aim of this research work is to reveal the effect of adding high modulus stainless steel and high toughness nano-silica along with pineapple fibre in fracture characteristics of epoxy hybrid composite. Both the pineapple fibre and wire-mesh were surface treated by using H2SO4 for better adhesion of reinforcements with the matrix medium. Epoxy hybrid composites were prepared using hand layup method followed by room temperature curing for 48 h. The tensile fatigue results revealed that the composite, which contains nano-silica of 1.0 vol% along with epoxy, pineapple fibre and wire-mesh in PF/SS/PF/SS arrangement gives the highest life cycle of 28520 counts. Similarly the composite, which contains nano-silica of 1 wt% along with epoxy, pineapple fibre and wire-mesh in PF/SS/SS fibre pattern (EFW22) gives the highest energy release rate of 0.86 MJ m⁻². The low-velocity drop load impact results show that the composite, which contains epoxy, fibre and wire-mesh in PF/SS/SS/PF/SS (EFW32) fibre arrangement with 1.0 vol% of nano-silica in epoxy resin gives very high penetration resistance and energy absorption compared to other composite designations. From strength factor calculations the composite made with epoxy, fibre, wire-mesh with PF/SS/SS/PF/SS stacking sequence along with 1.0vol% of nano-silica (EFW22) scores higher normalized strength of 98%. This high fatigue, fracture toughness and high penetration resistance against drop load epoxy composites could be an alternative material for automobiles, structural, surveillance mini air-crafts and domestic appliances manufacturing industries with high economic conditions.

1. Introduction
Natural fibre-reinforced thermoset composites are having unique applications in engineering such as automobiles, structural and other load-bearing applications [1]. Additions of natural fibres may convert the conventional polymer composites as eco-friendly materials since they are bio-degradable. Natural fibre composites are good enough in economical and cost-effective conditions at the same time strength wise they are marginally inferior [2]. On comparing with synthetic fibres such as glass, nylon, Kevlar the natural fibres offers low performance in load-bearing capabilities. Pineapple fibre could act as fibre reinforcement since it offers a superior tensile strength of 900 MPa with higher elongation of 1.2. It's density also relatively lower (1.3 g cm⁻³) compare with other natural fibres in its same class. To improve the load-bearing capabilities of natural fibre-reinforced composites ultra-fine micro metal meshes could be used along with natural fibres. These metal meshes could absorb the sudden and slow propagation load and offers high stability against plastic deformation of such composites. Stainless steel could act as wire-mesh reinforcement since they offer very high tensile
modulus, percentage of elongation, anti-rusting effects and cost-wise cheap. Karunakaran et al\cite{3} investigated the effect of adding acid-etched metal wire-mesh along with E-glass fibre-reinforced epoxy resin composite. The authors concluded that adding acid-etched stainless steel micro wire-mesh along with E-glass fibre improved the drop load penetration compare than composites, which is not-reinforced with micro metal mesh. Arun Prakash et al\cite{4} studied the effect of adding aluminium and stainless steel wire-mesh along with E-glass fibre in epoxy resin composite. The results were revealed that the additions of metal wire-meshes along with E-glass fibre greatly improved the tensile strength, flexural strength and also low-velocity drop load impact behaviour of composite. Addition of particle second phases along with fibre addition could improve the mechanical strength behaviour and thermal stability of composites. The micro or nanoparticle addition could greatly support in micro load sharing behaviour and helps in suppressing the micro-crack propagation in the matrix. Many engineering ceramics like B4C, Al2O3, Fe2O3 and SiC had utilized by many authors worldwide since these particles are high performance, eco-friendly, abundant in nature and cost-free \cite{5}. Among these particles, Silica could be a notable engineering ceramic particle because of its very low thermal expansion coefficient, non-soluble nature and easy preparation methods. Surface treatment of fibre, wire-mesh and particles could fetch significant improvements in mechanical, thermal and sudden load-bearing characters. The acid or base etched reinforcements could improve in adhesion phenomenon with base matrix via wavy nature of outer surface due to surface treatment process. The surface treatment process may create micro pits by leaching the surfaces through, which the resin could create physical bonding with reinforcements. Arun Prakash et al\cite{6} investigated the effect of acid and base treatment on glass-fibre and its effect on drilling characteristics. The authors confirmed that the acid treatment on fibre gives improved dimensional stability and low surface roughness on comparing with other surface-treatment methods. Moreover, the fibre orientation directly influences on mechanical and drop load impact behaviour of composite since the majority of load could transfer by the continuous fibre elements \cite{7}. Gokul et al\cite{8} studied the effect of adding E-glass and Kevlar fibre in three different laying patterns namely symmetric layering & aligned orientation, alternative layering along with alternative orientation and accumulated layering aligned orientation. The authors confirmed that the accumulated layering aligned orientation pattern fetched improved mechanical and drop load impact strength compare than other fibre laying pattern. Hand layup process could deploy to make layered epoxy composites since the process consume low power and low process parameters \cite{9}. In this process, wooden or metal moulds could be used with the wax-coated condition. The fabricated composite could cure at room temperature or some time at elevated temperatures. For better, mechanical and thermal properties post-curing is mandated since the quantum of cross-linking density directly proportional to the strength of the composite. The fabricated composite could test based on ASTM standards since these standards are unique and globally standardized. These mechanically strengthened and drop load damage resistance improved epoxy bio-composites could be used in structural, automobile, mini-aircraft manufacturing and domestic appliances manufacturing industries \cite{10}.

2. Experimental procedure

2.1. Materials

A quick set Araldite epoxy resin with density 1.2 g cm\(^{-3}\) and molecular weight 190 g mol\(^{-1}\) was utilized in this current study and procured from HUNTSMAN India, Pvt. The primary reinforcement pineapple fibre of density 1.3 g cm\(^{-3}\) and micro metal wire-mesh of density 7.8 g cm\(^{-3}\), the diameter of 200 \(\mu m\) were purchased form Go green India Pvt. Ltd The secondary addition nano-silica of size 20 nm was purchased from Sigma Aldrich, USA. The chemicals like H\(_2\)SO\(_4\) and ethanol for the surface-treatment process was purchased from MERCK, India, Ltd figure 1 shows the SEM and TEM images of both pineapple fibre, SS wire-mesh and nano-silica. Figure 2 shows the FTIR spectra of pure epoxy resin, pineapple fibre and nnao-silica particles. The observed peaks indicate the molecules present in each materials. A peak at 2900 indicates the presence of unsaturated O–H stretch on epoxy resin. Similarly, other strong peaks at 1500 and 900 indicates C–H bend, which is from long chain epoxy molecules. Similarly a peak at 3400 indicates the presence of amino group on natural fibre surface and peaks at 1900 and 14000 reveals the presence of C–H hydrocarbons present on fibre. From nano-silica FTIR peaks, a strong peak at 3200 indicates the presence of O–H (hydroxide) content and a peak at 1345 indicates the presence of O–stretch.

2.2. Acid etching of pineapple fibre and SS wire-mesh

The pineapple fibre and stainless steel wire mesh were surface treated by acid etchant H\(_2\)SO\(_4\) of 2N normality. In this, the concentrated H\(_2\)SO\(_4\) was taken in a wide-open beaker and the pineapple woven fibre and SS wire-mesh were briefly immersed in it for just 10 min. Since over timing may leach more in the wire-mesh and fibre the duration was limited to 10 min. The dipped fibre and wire-mesh was then separated from the acid solution and
Figure 1. SEM images of (a) SS wire-mesh, (b) Pineapple fibre and (c) TEM image of nano-silica particles.

Figure 2. FTIR spectra of pure epoxy; pineapple fibre and nano-silica.
cleaned with ethanol to remove the scales and dirt. The ethanol washed fibre and SS wire-mesh was then allowed to dry in room temperature for 1 h. Figure 3 shows the acid-etched pineapple fibre and stainless steel wire-mesh [4].

2.3. Hybrid composite fabrication
The fixed quantity of resin is mixed with 0.5 and 1.0 vol% of nano-silica particles and stirred continuously for getting a homogeneous solution. The resulted colloidal substance is poured into a silica rubber mould with a wax coating. Firstly a liberal coat of resin was applied then pineapple fibre and stainless steel wire mesh was laid based on the proposed laying pattern illustrated in figure 4.

The composites were cured at room temperature for about 24 h and post cured at elevated temperature (140 °C) about 48 h in a hot oven. The post-cured composites were taken out from the mould and checked for any visual defects and designated as narrated in table 1.

2.4. Characterization
The post cured epoxy hybrid composites were machined for further evaluation processes. The samples were cut using an abrasive water jet machine (WATERJETS, 9000, Germany) having the flow velocity of 220 MPa, an abrasive flow rate of 0.3 g s⁻¹ and nozzle diameter of 1.1 mm. The test samples were machined in-accordance with ASTM standards. The tensile fatigue properties of epoxy composites were tested based on ASTM D 3479 using a tensile-tensile fatigue machine (MTS Landmark 370 load frame, USA). The stress ratio was maintained as 0.1 with a loading frequency of 5 Hz. A maximum load of 1 KN (around 50% of maximum tensile load), the elastic modulus of 6.0 GPa and working ambience of 250C were maintained as process parameters. The fracture energy release rate and plane strain measurements were taken in-accordance with ASTM D 5045 using a universal testing machine. The local driving force for crack extension measures through stress intensity factor (Kic) with MPa/m as a unit and the energy release rate is denoted as G1c with a unit of MJ m⁻². Similarly, the drop load impact behaviour was evaluated using a hemispherical dropper of diameter 10 mm (INSTRON-9000, UK) in-accordance with ASTM D-4762. The impactor mass of 2 Kg and indenter velocity of 3 m s⁻¹ was set as process parameters. The test was repeated at least 5 times to compute average. The sub-micron images were
3. Results and discussion

The various properties and overall goodness of fabricated composites were analyzed using strength factor method. This strength factor was calculated by computing the normalized strength factor concerning the highest value for each property in each composite. Finally, the average of all individual normalized strength was calculated.

3.1. Tensile fatigue behaviour

Table 2 shows the tensile fatigue behaviour of pure epoxy resin and its composites. The pure epoxy resin gives a fatigue life cycle of just 650. This lower fatigue life counts are the reason for the absence of microcrack suppressing mechanisms within the matrix. When a repeated load is applied on the material the residual stress are accumulated in the brittle nature of the epoxy resin. These stresses create high-stress intensity factor, which propagates the micro-cracks and led the epoxy resin to plastic deformation \[11\]. But it is noted that the additions of pineapple fibre and stainless steel wire-mesh along with nano-silica increased the fatigue life. The different fibre and wire-mesh pattern fetched a remarkable change in fatigue behaviour composites. The lowest fatigue life cycle of 24410 is observed for composite designation EFW11. The addition of acid-treated pineapple fibre and metal wire-mesh greatly support in higher fatigue life. The wavy pattern of both pineapple fibre and metal mesh increased the adhesion betweenibre/metal mesh to the matrix due to the self-locking mechanism by the presence of wavy surface and micro pit marks. Further addition of nano-silica of 1.0 vol% in the same designation improves the fatigue life cycle. This improvement is because of adding more volume percentage of nano-silica reduces the microcrack development and suppress the crack propagation further. The addition of nano-silica effectively bond with the epoxy matrix and fills in the voids of the matrix and reduces the crack propagation \[12\ and 13\]. A highest fatigue life cycle of 28520 counts is noted for composite designation EFW32. This high fatigue strength is because of alternate layering of fibre and metal wire-mesh along with 1.0 vol% of nano-silica.

The presence of natural fibre on the outer side significantly reduces the stress formation on the outer surface, which led the composite to withstand for higher fatigue life. It is noted that compare than 0.5 vol% the 1.0 vol% of nano-silica gives much-improved fatigue life cycle. This improvement is because of effective bonding and void filling by the presence of nano-silica in the resin matrix. Thus the additions of acid-treated pineapple fibre, stainless steel wire mesh with alternate layering and presence of nano-silica (1.0 vol%) offers larger fatigue life count. Figure 5 shows the fractography of composite designations EFW11 and EFW32. The images revealed that formation of more micro-cracks are the cause of plastic failure at the lower life cycle and in composite designation EFW32 the micro-cracks are lesser and no interfacial cracking was found. The presence of shear cups indicates that the composite holds improved ductility and very high adhesion \[14\]. This improvement is because of the presence of a larger volume of nano-silica, which bonded with matrix tightly and reduces the crack propagation. This phenomenon reduces the interfacial delamination and making the composite to withstand for better-repeated loading.

| Material designation | Epoxy (vol%) | Fiber + wire-mesh (vol%) | Nano-silica (Vol%) |
|----------------------|-------------|-------------------------|-------------------|
| E                    | 100         | 0                       | 0                 |
| E FW11               | 59.5        | 40                      | 0.5               |
| E FW12               | 59.0        | 40                      | 1.0               |
| E FW21               | 59.5        | 40                      | 0.5               |
| E FW22               | 59.0        | 40                      | 1.0               |
| E FW31               | 59.5        | 40                      | 0.5               |
| E FW32               | 59.0        | 40                      | 1.0               |
| E FW41               | 59.5        | 40                      | 0.5               |
| E FW42               | 59.0        | 40                      | 1.0               |

E-epoxy; F-fibre; W-wire-mesh.
### Table 2: Strength factor of composites

| Composite designation | Tensile fatigue (Cycle) | Normalized tensile fatigue (%) | Fracture toughness (MPa) | Normalized fracture toughness (%) | Energy release rate (MJ/m²) | Normalized energy release rate (%) | Drop load energy absorption (J) | Normalized drop load energy absorption (%) | Strength factor (%) |
|-----------------------|-------------------------|--------------------------------|--------------------------|-----------------------------------|-----------------------------|----------------------------------|-------------------------------|---------------------------------------------|-------------------|
| E                     | 650                     | 3                              | 9.3                      | 15                                | 0.15                        | 16                               | 0.3                           | 1.6                          | 9                 |
| EFW11                 | 24410                   | 86                             | 41                       | 66                                | 0.66                        | 77                               | 15.3                          | 81                           | 78                |
| EFW12                 | 25280                   | 87                             | 48                       | 77                                | 0.75                        | 87                               | 14.2                          | 76                           | 82                |
| EFW21                 | 25755                   | 90                             | 52                       | 84                                | 0.71                        | 83                               | 13.6                          | 72                           | 82                |
| EFW22                 | 26320                   | 92                             | 57                       | 92                                | 0.78                        | 91                               | 14.2                          | 76                           | 82                |
| EFW31                 | 27632                   | 97                             | 55                       | 89                                | 0.80                        | 93                               | 18.8                          | 100                          | 95                |
| EFW32                 | 28520                   | 100                            | 62                       | 100                               | 0.86                        | 100                              | 17.4                          | 93                           | 98                |
| EFW41                 | 26845                   | 94                             | 51                       | 82                                | 0.78                        | 91                               | 16.8                          | 89                           | 89                |
| EFW42                 | 26170                   | 92                             | 57                       | 92                                | 0.86                        | 100                              | 17.0                          | 90                           | 94                |

E-epoxy; F-fibre; W-wire-mesh.
3.2. Fracture toughness

Table 2 shows the fracture toughness and energy release rate of pure epoxy resin and its composite. It is observed that the pure epoxy resin gives a very lowest fracture toughness of 9.5 MPa and the energy release rate of 0.15 MJ m$^{-2}$. This lower value in fracture toughness and energy release rate is the cause of no strengthening mechanism built-in brittle epoxy matrix [15]. It is observed that further addition of pineapple fibre and stainless steel wire-mesh along with nano-silica improved the fracture toughness and energy release rate. The composite designation EFW32 contains alternate layering of fibre and metal mesh with 1.0 vol% of nano-silica offers the highest fracture toughness of 62 MPa and energy release rate of 0.86 MJ m$^{-2}$. This is near 11% of improvement when compared with 0.5 vol% of nano-silica in its similar class. This improved value is the reason of effective load sharing phenomenon of fibre and metal mesh. When tear force is applied on the composite the presence of fine fibre elements receives and share the load effectively [16]. The presence of metal wire-mesh also receives and shares the load due to its continuous pattern and reduces the stress intensity factor on the matrix. The acid etched ultra-fine dimension of steel wire-mesh improved in adhesion and chemical affinity with matrix due to the wavy nature of its surface and micro pits. When laying the acid-etched metal wire-mesh in the epoxy resin the resin is entering into the micro pits and creating a strong physical bond. This nature improves the load sharing ability of the matrix to reinforcement and reduction of stress intensity factor [17]. And also the improved adhesion reduces the interfacial cracking, which is developed due to microcrack development and its propagation along the interfacing zone. Figure 6 shows the fractured specimens after fracture toughness test. It is visible that the acid-etched metal wire-mesh shows improved adhesion. The surface of the wire-mesh contains the debris of matrix after applying the tear force and the matrix interface is broken finely without any pull-out phenomenon, which explicates the adhesion improvement between fibre/metal wire-mesh to epoxy matrix.

It is observed that in all composite designations the large volume of nano-silica (1.0 vol%) is seen giving better results than 0.5 vol%. This phenomenon is because of more load-sharing elements in the matrix medium and enhanced adhesion nature of nano-silica particles with epoxy resin. When large volume particles are placed
in the molecular voids the load will be uniformly shared without any interruption. The improved tear modulus and suppressing of crack propagation led the composites to become tougher one [18].

3.3. Low velocity drop load impact behaviour

Table 2 gives the low-velocity impact behaviour of pure epoxy and its hybrid composites. The energy absorption behaviour of pineapple fibre and metal wire-mesh reinforced epoxy composite is much better than pure epoxy resin. Maximum energy absorption of 18.8 J is observed for composite designation EFW13, while the pure epoxy resin gives only 0.3 J. This massive improvement is the reason of effective load blocking and receiving fibre and high modulus stainless steel wire mesh. It is observed that the alternate layering of pineapple fibre and metal wire-mesh fibre pattern EFW31 gives highest penetration resistance than other composites. The impactor indentation is not seen on the penetration side, which explicates the effective penetration behaviour of hybrid composite EFW31. It is observed that the hybrid composites, which are made up of stainless steel wire mesh as top layer giving marginally less impact energy absorption than those composites made of pineapple fibre as a first layer. The low energy absorption of 15.3, 14.2, 13.6 and 14.2 J was observed for composite designations EFW11, EFW12, EFW21 and EFW22 respectively. This discrimination between composites is because of improved ductility on the hybrid composite surface, which is made of pineapple as the first layer. When the high fibrous pineapple fibre facing the sudden load, the load is uniformly shared due to improved ductility [19]. But the composite, which contains micro wire-mesh as the top layer, the matrix phase get plastic deformation quickly and widespread. This is because of the fibrous pineapple fibre finely mixed with matrix phase and completely receives the incoming sudden load. It is observed that the composite designation EFW31 and EFW32 alone giving marginally higher energy absorption because in this composite designations alone the pineapple fibre is layered in the top portion. Figure 6 shows the drop load impact damage characteristics of hybrid composites tested. The advancing side and penetrating side of each composite are illustrated in figure 7.

From figures, it is well understood that the composites, which is made up of metal mesh as the top layer are giving random fracture due to high brittleness. It is well said by a research team Arun Prakash et al [4] about the effect of OH functional group reaction with metallic wire-mesh surfaces and development of aqua molecules along with the metal mesh-resin interface. These formed aqua molecules weaken the interfacial strength and induce plastic deformation on the composite at lower loads itself. But the composite, which contains pineapple fibre as top layer no such effects are created hence the top layer finely receives the load and transmit it to subsequent layers. Even though in composite designations EFW31 and EFW32, the subsequent layers are built with stainless steel wire-mesh the composites are with-stand for high load and high energy absorption. This effect is because of when the sudden load reaches the metallic surface its impact is less and not creating much effect on the composite strength. Thus improved drop load impact behaviour is observed in composite designations EFW31 and EFW32. It is observed that the impactor created almost 3/4th of penetration depth in these two composites (EFW31 and EFW32) and no such deformations found in the rear end of composites whereas other composites gave blind open holes. This effect is because of effective impact load suppressing behaviour of composites with primary and secondary reinforcements. Firstly the wire-mesh absorbs maximum load and reduces the intensity of applied force, then the presence of pineapple fibre gradually disperse the load throughout the matrix and reduces the stress concentration factor [20 and 21].

Second, the presence of nano-silica particles in epoxy matrix effectively bond with resin and suppress the micro-crack propagation since the micro-cracks are the root cause for plastic deformation in any composites. The presence of nano-silica also absorbs aqua molecules, which are generated during the curing process and maintains the composite more stable under external sudden load conditions. The addition of nano-silica also provides very high adhesion between reinforcement and matrix. Thus the interfacial delamination is typically arrested and leading the composite tougher [22].

It is observed that compared with composite designation EFW32 the composite EFW31 gives marginally higher result in energy absorption and penetration resistance. This is because of a larger volume (1.00 vol%) the nano-silica particles are possibly agglomerated and increase the stress concentration [23]. Thus relatively lower energy absorption found. Moreover, the difference between EFW31 and EFW32 is not much higher and it is clear that addition of nano-silica greatly influences in low-velocity drop load impact behaviour. Figure 8 shows the numerical simulation format of composite with impactor. The test was performed using a Hyper-mesh and LS Dyna with recommended inputs from previous tests. Figure 9 shows the delamination and plastic failure of the hybrid composites. It is observed that in EFW1 the impact damage is very high and travelling towards both traverse and axial direction. Similarly, the other composites also giving relatively larger deformation like EFW1. This larger deformation is the cause of absence of load bearing and load distribution mechanisms are available. It is observed that the composite designation EFW21 and EFW22 giving considerably larger impact damage. This impact is because of no energy absorption elements within epoxy matrix along with fibre. The presence of SS/SS/KF/KF fibre pattern produced larger damage on the top surface because of improved brittleness on the
surface by the addition of wire-mesh. It is observed that the composite, which is made up of KF/SS/KF/SS fibre stacking sequence along with nano-silica of 1.0 vol% giving excellent resistance against drop load. There are no such heavy damages on the surface and the vertical penetration was almost arrested. The rear end impact damage

Figure 7. Drop load impact damaged hybrid composites.

Figure 8. Hyper-mesh LS Dyna impact model.
reveals no such damaged counter-parts are appeared, which reveals very high penetration resistance of composite. The presence of kenaf fibre on the top layer highly miscible with resin and equalize the load in all direction. The equalized load is not creating much damage on the impacting surface and penetration depth too. Thus the addition of wire-mesh into epoxy resin with significant manner (KF/SS/KF/SS) yields notable improvements in impact damage analysis [24].

4. Conclusions

The epoxy hybrid composites were fabricated using pineapple fibre, stainless steel micro wire-mesh and nano-silica and characterized successfully. Based on the results the following conclusions could be made.

(1) The surface-modification using H₂SO₄ process significantly improved the adhesion behaviour of reinforcements to matrix.

(2) The tensile fatigue results revealed that 1.0 vol% of nano-silica dispersed epoxy resin with alternative layering of pineapple fibre and steel wire-mesh, in this pineapple fibre as top layer.

(3) Similarly the highest fracture toughness and energy release rate is observed for composite designation EFW₁₂, which contain symmetric alternate layering of pineapple fibre and steel wire-mesh.

(4) The drop load impact behaviour of composites revealed that composites, which contain EFW₁₁ and EFW₁₂ gives very high penetration resistance against sudden loading, since the wire-mesh on the top layer in

![Figure 9. Shows the load-distribution analysis on composites.](image)
layering pattern gives poor values in low velocity drop load impact behaviour. This claim has been confirmed using numerical simulation with load vs. distribution.

(5) The SEM fractograph images of fatigue, fracture toughness and drop load impact toughness of composites shows improved adhesion of reinforcements with matrix by the addition of nano-silica.

(6) Over all from the strength factor, the composite designation EFW32 produced maximum strength factor as 98% hence this proposed model would give overall best result.

(7) These fatigue strength and fracture toughness improved high drop load penetration resistance epoxy composites could be used in structural, automobile and other industrial applications.

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ORCID iDs

T Dinesh @ https://orcid.org/0000-0003-2964-7708
A Kadirvel @ https://orcid.org/0000-0001-9314-9311

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