Analytical nondestructive evaluation for materials characterization

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Science and technology of nondestructive testing and evaluation has contributed immensely to the safety and productivity of industrial plants. In recent years, nondestructive evaluation (NDE) has emerged as a frontline research area of equal if not greater technological relevance, for materials characterization as well. A comprehensive range of techniques from qualitative nondestructive testing for quality control of engineering products and materials to quantitative NDE for materials characterization is being used by the engineering industry and materials researchers, for better understanding of the manufacturing practices and materials behaviour. Quantitative NDE is considered essential for ensuring fitness for purpose at the start of the life in case the component has been designed using fracture mechanics parameters. Quantitative NDE is also vital for assessing degradation of material during service. Moreover, quantitative NDE enables characterization of dynamics of certain phenomenon (not achievable by destructive test methodologies) leading to better understanding of the performance of materials in relation to unavoidable defects in the materials.

As the next logical step, the need for an analytical approach to NDE is felt. The need and motivation for such an approach is addressed and the means to achieve this objective are identified. It is argued that analytical NDE is essential to meet the challenges of characterization, intelligent processing of materials and life prediction of components and plants. These requirements are of significant importance in the context of recent developments in materials engineering, and for enhancing the competitive advantage of Indian engineering industry in the international market.

NONDESTRUCTIVE evaluation (NDE) is meant to characterize the material, component and plant with the objectives of assessing the performance and better understanding of the behaviour.

NDE should not impair, in any way, the usefulness of the test object for the intended performance level. NDE techniques are based on a wide range of physical principles derived from the understanding of the interactions of electromagnetic radiations (optics, X-rays, etc.), ultrasonics, electric and magnetic fields etc. with materials. The exploitation of these techniques is made possible due to developments in engineering fields especially sensors, instrumentation, automated devices, computers, etc. The sensitivity of these techniques covers a range of a few hundred angstroms to a few millimetres. The materials amenable to evaluation are metals and alloys, composites, ceramics, plastics, etc. The components can be as small as an integrated circuit or a large pressure vessel, an integrated pipeline or a complete plant.

Science and technology of nondestructive testing and evaluation has contributed immensely to the safety and productivity of industrial plants. In recent years, NDE has emerged as a frontline research area of equal if not greater technological relevance, for materials characterization as well. In the early days, most of the NDE concepts were qualitative, with the aim of arriving at the acceptance or rejection decision with respect to specified criterion. It is now fully realized that complete materials characterization requires not only qualitative, but also quantitative NDE. Quantitative NDE should characterize the materials and defects with the requisite numbers and spatial information. This would enable relating these characterization numbers to the performance levels. Quantitative NDE also enables characterization of dynamics of certain phenomena (not achievable by destructive test methodologies), thus leading to better understanding of the performance of material in relation to unavoidable defects in the materials.

Science has characteristics of dynamism and evolution. An elegant idea, after germination, may find a safe niche to grow, with support in the early years from the existing engineering and technology. As time goes on, the idea, if developed to a reasonable extent, comes under the critical scrutiny of science and gets accepted or rejected based on its ability to withstand competition and contribute to scientific knowledge and technological demands. Finally, a state of self-sustaining growth is reached wherein the pace of growth is dictated by richness of science and the technological fall-outs. And, this is a continuous process. The growth in any challenging and exciting discipline such as NDE is governed by the pushers and pullers. Pushers are the demand on the relevant science and technology. Pullers are the available inputs which help the discipline to meet these demands. Table 1 gives the 'pushers and pullers' as applicable to NDE.

Table 1. Pushers and pullers of NDE science and technology

Pushers	Pullers
Life prediction	Automation
Residual life assessments	Smart sensors and instrumentation
Characterization of new materials	Signal and image analysis
Productivity enhancement	Artificial intelligence
Reduction of scrap	Experience coding in the form of expert systems
Quality production with minimum inputs	Basic research

Levels of NDE

Qualitative NDE

A qualitative approach to NDE attempts to report testing with little or no measurements made to substantiate its decisions related to quality or performance. With this view in mind, we can say that early approaches to NDE were largely qualitative.

For qualitative NDE, acceptance standards are available and the expected technology level from this approach is generally achievable. It uses appropriate sensors and first order instrumentation just enough to describe the phenomenon under observation. This sort of an approach, is still practised in many industries and shop floors. However, it is realized that this approach does not allow full benefits to be derived from NDE.

Quantitative NDE

A quantitative approach in NDE describes the measurements precisely, and also substantiates the descriptions *numerically*, with adequate spatial information. It removes subjectivity from interpretation of test results. Smart instrumentation, and better understanding of the interaction of testing medium with the test object enables performing quantitative NDE.

Standards are being evolved for quantitative NDE approaches. Quantitative NDE is increasingly being used for the challenging engineering endeavours in nuclear, space and defence industry routinely, though its impact on conventional engineering industry is just beginning. A typical case of quantitative NDE is the classification of defects, size and shape distribution of defects, microstructure description, etc. with measureable physical quantities. Such an approach needs appropriate sensors, advanced instrumentation and adequate signal processing and analysis.

With the advances in sensors, electronics, instrumentation and computer technology, more and more conventional NDE techniques are being pushed to quantitative realm. Examples are C-scan and P-scan ultrasonic techniques, and acoustic microscopy.

Analytical NDE

Analytical techniques in NDE should be able to correlate results with analytical models and predict measurements for a given defect or feature. Quantitative NDE, to qualify for the analytical NDE level, must satisfy the essential requirements of predictability of the results based on scientific principles. Tomography, 3-D modelling for prediction of eddy current signals from the defects, residual stress determination by X-ray diffraction, synthetic aperture focussing technique for ultrasonic imaging of defects, etc., are a few of the analytical techniques in NDE, which have matured from laboratory development stage to technologically relevant applications. No standards or codes are available, at present for analytical NDE.

The keyword in qualitative approach is 'description'; that in quantitative approach is 'measurements'; and in the case of analytical approach they are 'modelling' and 'predictability'.

Analytical NDE: Challenges and approaches

We have witnessed a spurt in the NDE activities during the recent past. The significant growth in NDE is fuelled by the challenges, namely characterization and testing of new materials, stringent testing and evaluation requirements and demands for the application of NDE to life prediction and intelligent processing of materials. Computer, robot and sensor technologies coupled with advances in modelling towards understanding of physical interactions of testing medium with material and component, are the prime resources which have helped NDE professionals to meet these challenges.

There is an urgent need in most of the disciplines and particularly in NDE, for providing cost-effective and reliable solutions to challenging engineering problems. The rapid replacement of existing materials with newer materials in the industry offers no time for empirical understanding of these materials, thus neccessitating an analytical understanding of these materials. This necessity is paving the way for the entry of advanced concepts. As the demands become stringent in terms of sensitivity and acceptability criteria, recourse to analytical NDE methods is only logical, since most of the 'conventional' NDE approaches do not satisfy the new challenges posed to NDE professionals.

The realistic problems mentioned above have very limited solutions at the present development level of NDE science and technology. This prime necessity is an impetus to the high pace of the interdisciplinary research and development efforts in NDE.

It is clear that for solutions to the problems outlined above, universal standards with artificial defects (as conventionally practised) are invalid and illusory. This observation is based on the fact that many individual factors can simultaneously influence the 'sensor' used to interrogate materials for assessing their microstructure and, morphological and mechanical property variations. Sophisticated interpretational methods are required to extract desired information from the signals, with respect to material properties or characteristics. Thus advanced signal and image analysis, pattern and cluster analysis, artificial intelligence and mathematical modelling are the technologically relevant areas which need to be harnessed extensively for meeting these present and future challenges with confidence and reliability.

The reasons for slow adaptation of quantitative and analytical approaches in NDE are manifold. Firstly, the inverse problem in NDE has received little attention. The inverse problem, i.e. to model the (acoustic, electric or electromagnetic) wave–material interaction on the basis of the observed features in the signal space such as frequency components, rise time, transfer functions, spectrum broadening, preferential attenuation, etc. is a phenomenological investigation that requires extensive mathematical modelling. Secondly, the established codes and practices used worldwide have not yet supported or made mandatory the use of these advanced techniques. However, it should be mentioned that not all situations may warrant the use of an analytical technique for testing and evaluation. Technological drives and demands for stringent quality assurance and improvement at all stages in the life of a component, are the motivating factors pushing the research in the area of inverse problems and analytical NDE.

As scientific pursuits diversify and grow, newer tools for the growth are discovered and invented. Mathematics is the universal tool. It offers a medium of communication and analysis of scientific knowledge precisely. Depending upon the requirements, every segment or category of scientific knowledge makes use of one or more of the tools. NDE is no exception. For NDE, apart from mathematics, the new members in this array of tools are signal and image (pattern) processing and analysis, cluster analysis and control and information theories and artificial intelligence, etc., or in short cybernetics*. In this section we shall explore how these tools aid in elevating NDE to an analytical status.

Mathematical modelling in NDE

Basically, NDE involves the interrogation of a material or a component with a medium and studying its outcome. This medium could be either active (ultrasonic waves, eddy current, laser, etc.) or passive (acoustic emission, Barkhausen noise, etc.). If it is possible to understand the interaction, these media have with the material/component, then we would be at a greater advantage in characterizing the material/component to the fullest possible extent.

Such an understanding is possible if we can enunciate a model of interaction and verify it experimentally. In such a model, the variables could be the material/component themselves, their physical (and possibly chemical) properties, shapes and sizes of defects, etc. apart from those of the interrogating media. In order to obtain complete understanding, each of these variables must be studied in depth on their own merit and in relation with the changes in the other variables. This is easier said than done. The relation between the input and the output signal is of immense importance in gaining understanding about the medium (microstructure, material properties, etc.) and defects in the medium (their type, size, location, shape, orientation, etc.). The essence of the problem and the various stages involved in achieving this goal are shown in Figure 1. Ways and means to realize this objective are also shown in Figure 1 and are described in detail in the following paragraphs.

In order to handle and study these variables, mathe-

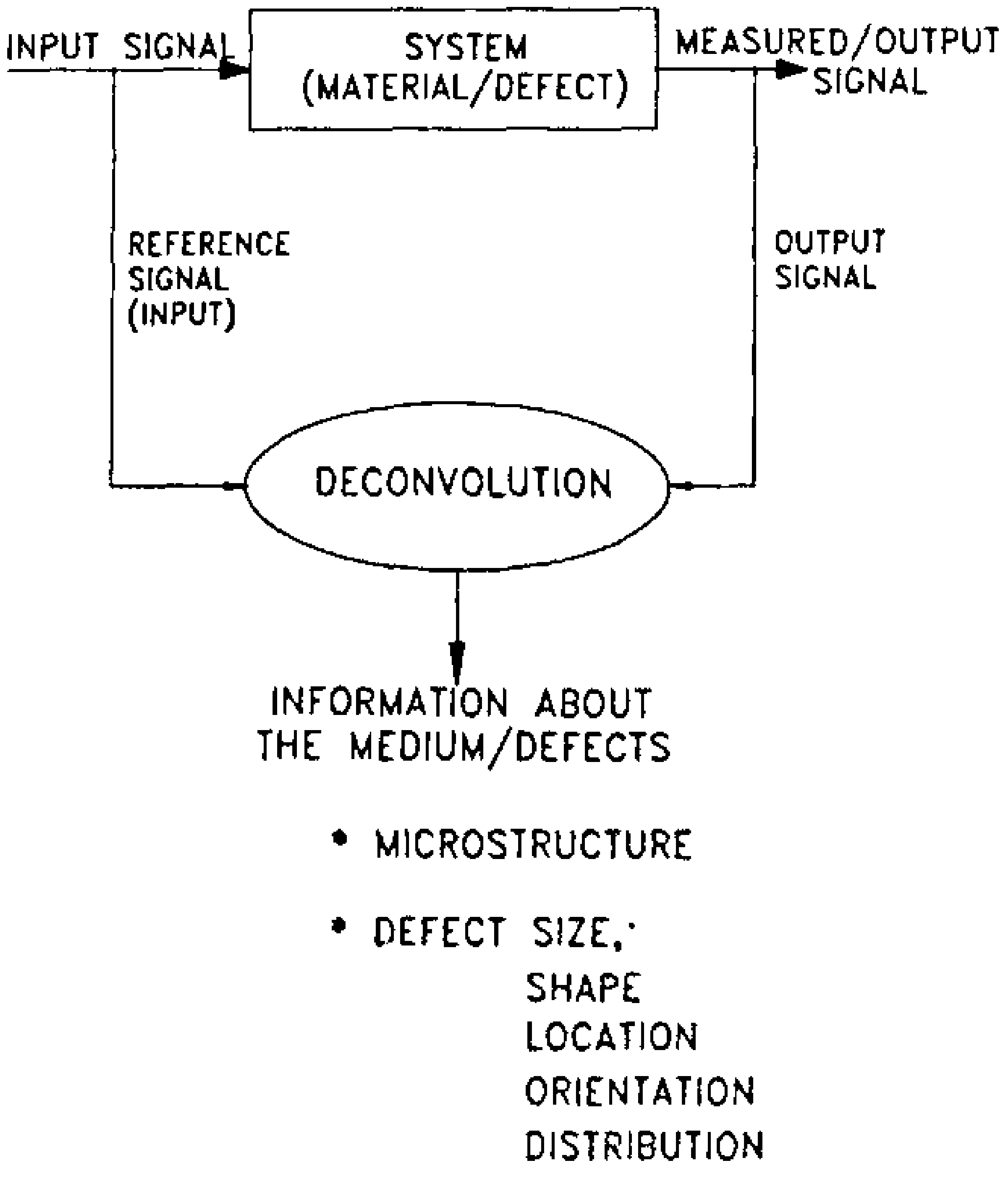

Figure 1. Schematic representation to obtain material/defect characteristics.

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*Cybernetics is a term that denotes the study of control and communication in, and particularly between, humans and machines and comprises automation, robotics, artificial intelligence, information theory, bionics, automata theory, pattern and image analysis, control theory, signal processing and analysis, heuristics, etc.[1].

matical tools are essential as is the deep understanding of the physical principles involved. A few such studies have been reported[2-4], but since the use of newer materials is ever increasing, it can be confidently stated that such phenomenological studies should be pursued vigorously and are fertile areas of research in NDE.

Signal and image processing and analysis in NDE

Once a phenomenon is modelled, it is to be verified and validated to make it acceptable. One of the ways to test a model is to interrogate the material using a variety of sensors and to analyse the outcoming signal from the material. The use of complementary sensors sensitive to specific characteristics of the material being studied and exploitation of a variety of signal analysis approaches provide the possibilities for the solution to the inverse problem with the help of phenomenological models. This is to be followed by correlating the features of the outcoming signals to the physical phenomenon taking place in the material. Finally, the result of this study and its deviation from the outcome of the model serves as a feedback in refining the model. Figure 2 shows the steps involved in such a study.

Signals carry valuable information. Extraction of the desired information hidden in a signal and the presentation of this information, in a way that is useful, is a major concern in signal analysis. Poor signal-to-noise ratio (SNR) makes the problem of extracting useful information formidable and challenging. Digital signal processing and analysis is not only used in situations where SNR is very poor, but also when it is required to take automated decisions, analyse large amounts of data and analyse data in real time. Also, signal and image processing methods serve as a useful interface between the abstract model that is being studied and the real experiments that are being performed to validate the model.

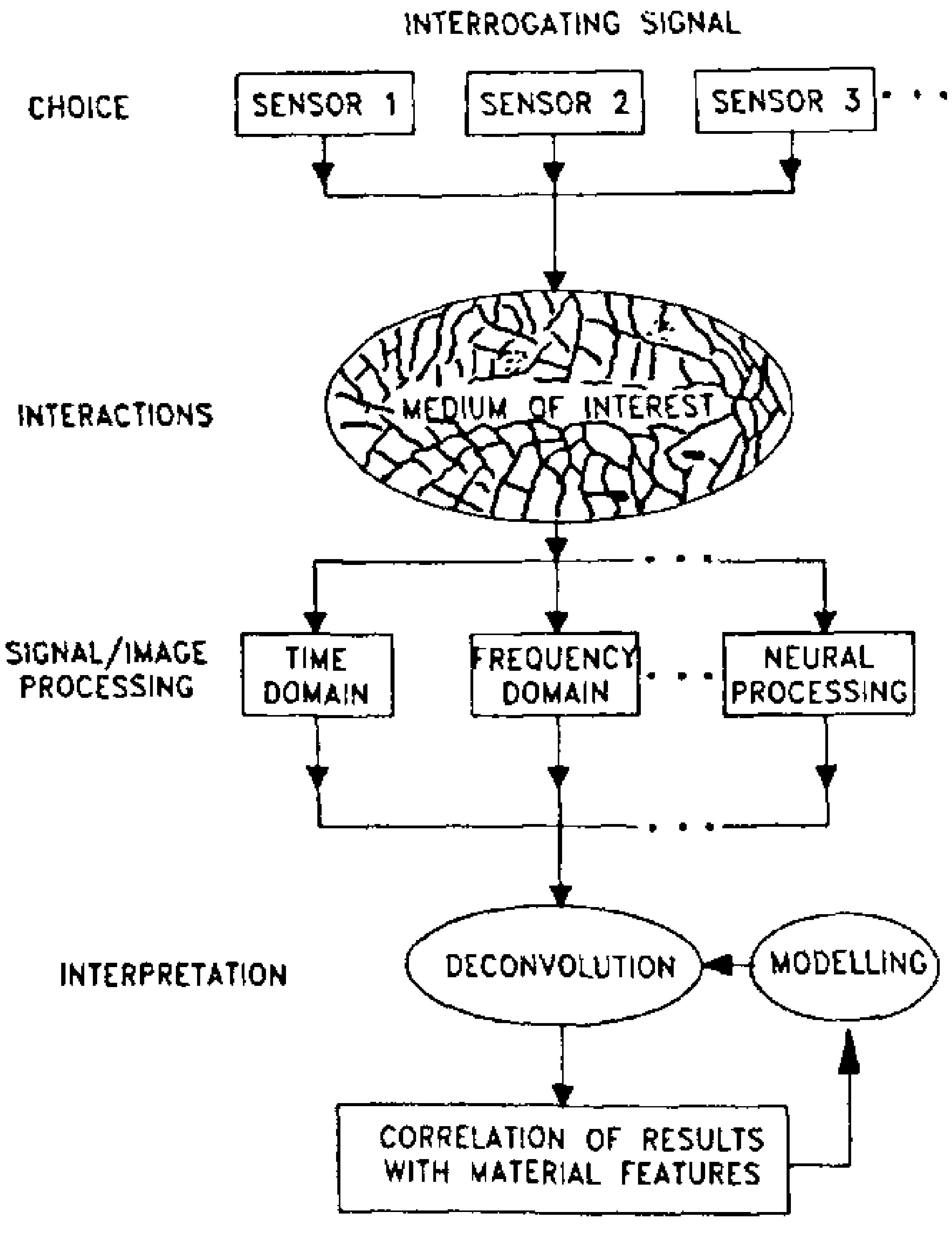

Figure 2. Steps involved in the inverse problem.

The advantages of using the appropriate signal and image processing techniques in conjunction with the conventional testing techniques are manifold. Apart from repeatability, these open an avenue for storage and easy and fast retrieval of test data not to mention the added advantage of the possibility for real-time data processing[5]. Signal analysis is now being applied to nondestructive testing not as a complementary technique but as an essential and useful extension of the present test procedures.

Artificial intelligence in NDE

The design, development and application of artificial intelligence (AI) in the field of analytical NDE in the coming years is expected to be influenced by a variety of factors. NDE is essentially an interdisciplinary area. Hence it involves the use of a variety of concepts drawn from physics, chemistry, materials science, instrumentation and mechanical engineering. The need to elevate NDE as an analytical science adds mathematics to this list. However, unlike other areas in science, the present day NDE more often involves subjective interpretation of results, involving a wide variety of factors (variables). This unique combination of *multiplicity of variables* involved and the use of possibly *incomplete and imprecise data* (*subjectivity*), makes NDE a potential user of advanced AI concepts that deal with large amounts of subjective data, both verbal and numerical. Hence, AI concepts are expected to serve a managerial role in the elevation of NDE to analytical status, i.e. one that keeps track of everything and informs the user about the right aspect at the right time.

The need to identify and apply the right technique for every situation/material/component, is a major factor, in the success of NDE. For a cost-effective solution and to save time and manpower, appropriate NDE technique will have to be recognized for each problem. Some possible scenarios, where AI concepts could aid NDE, are described in the following paragraphs.

The need to effectively use the accumulated knowledge about the NDE technique being applied, is an important factor that will accelerate the use of AI for NDE. For example in ultrasonic testing (UT) of welds, proper selection of the UT method, probe, etc. will solve a major part of the problem. Such complex tasks can effectively be performed by expert systems (ES), which are demonstrable programs that use AI concepts. An ES is 'ideally suited (a) when problems cannot be well-defined analytically, but when the number of alternate solutions is large, (b) the domain of knowledge is vast, and (c) relevant knowledge needs to be identified rapidly, as it is to be used selectively'[6].

The need for real-time monitoring of the process parameters/materials characteristics, as and when the material/component is being manufactured, is being realized all over the world. The emphasis of NDE will shift from finished product testing/evaluation to, testing/evaluation of the product as it is being manufactured. Definitely, this will involve monitoring and rendering advice on the behaviour of a multitude of process parameters. This is a case where a range of complex or large tasks are to be completed quickly, where instead of a single main software program, a group of subroutines may have to interact with each other to produce the desired results. The idea of using a number of separate software programs with common objectives, to work together to solve large complex problems, is the essence of distributed AI[7].

The need to find effective methods to store and retrieve NDE data, and the history of the testing/evaluation of all the components, is strongly felt by the personnel managing large industrial plants. This is required in order to record details such as date of testing, method of testing, name of the component being tested, operator details, test results, change in test results during subsequent testing/evaluation, variables of the model that are being changed, possible feedback, earlier remedial actions, conclusions, suggestions for future-tests, etc. The knowledge and databases pertaining to these aspects will be very large, but the expertise to assimilate and apply this large amount of knowledge at every stage of the production and NDE of materials and components, will be scarce. Intelligent and timely application of this knowledge in the production and NDE of manufactured materials and components, would be the key for success in our industrial ventures. There is already an acute and growing shortage of human experts in NDE and quality disciplines. This aspect drives the efforts towards adopting such of these operations which result in cost savings and better quality. Expert systems are expected to play a mature and knowledgeable advisory role in this aspect.

From the above-mentioned discussion, it can be inferred that a pragmatic and intelligent combination of mathematical modelling and cybernetics, as mentioned earlier, will have a major and important impact on the growth of NDE in the immediate future. A combination of these two areas of science will aid in a big way in elevating NDE to analytical status.

Figure 3 schematically shows the interplay between the various tools to be used in analytical NDE. Communication between the modules/tools takes place along the three buses, viz. data bus (numerical data), features bus (features of the signals) and knowledge bus (knowledge transactions between the various tools and feedback from the tools to the end user).

Any NDE inspection involves sensors and certain instrumentation. Output from this inspection is in the form of signals that require some initial processing, after which it is fed to visual examination module, signal analysis module, image analysis module, etc.,

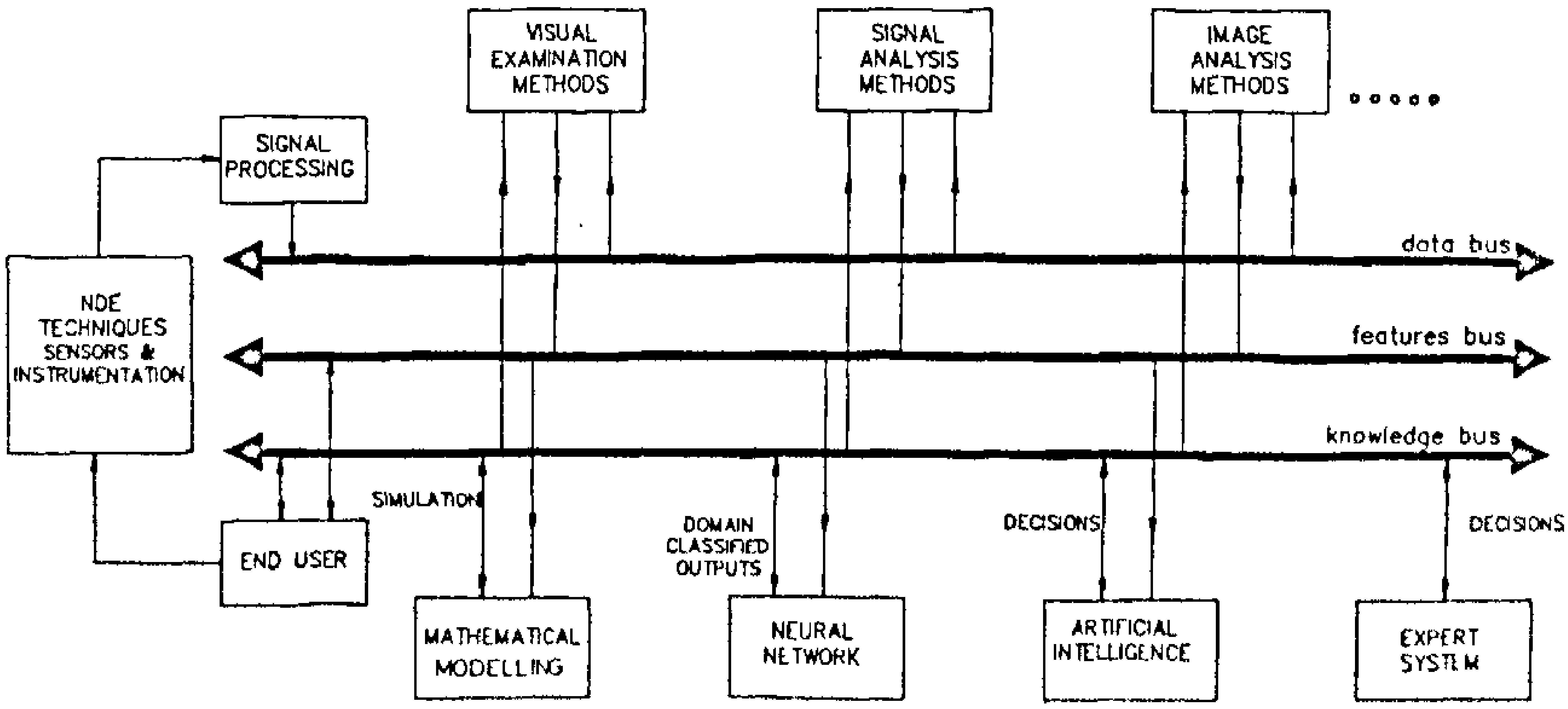

Figure 3. A future perspective of NDE.

through the data bus. Outputs from these modules are in the form of observed and measured features/patterns, which are fed via the feature bus to advanced modules such as mathematical modelling, artificial neural networks and artificial intelligence modules. These modules use and process the features/patterns to elicit knowledge, which is fed to the knowledge bus. This knowledge is used by the end user, connected to the knowledge bus, to achieve analytical NDE and, to understand the processes involved and to make corrections in the methodologies adopted, if necessary. The knowledge bus also interacts with other advanced modules, if necessary. The end user will use the expert system as the front end of this scheme, which will coordinate all the functions and bus traffic among all the advanced modules.

Analytical techniques in NDE—A bird's eye view

As mentioned earlier, some of the NDE techniques have evolved to gain the analytical status. In this section, we shall dwell upon a few examples of analytical NDE techniques of wider significance.

Computed tomography

Computed tomography (CT) is the computer reconstruction of the internal cross sectional structure of an object from a finite number of multi-angle projections obtained using penetrating radiations.

CT determines, quantitatively, point by point density values in thin cross-sections of a test object. A good quality CT using high performance linear array detectors, can differentiate densities with a resolution of 0·1% to 0·2% (approximately two orders of magnitude better than with film radiography). CT images are free from superposed features, and structural noise (a disadvantage in projection radiography) is absent. Thus although the spatial resolution of CT images is intrinsically less than the conventional radiographs, the effective information content is much higher.

CT can also provide detailed physical information about the defect with unparalleled clarity regarding its interpretation. This information (size, shape, location, density, etc.) can be used for quantitative evaluation.

In ordinary radiography, a shadow of the three-dimensional (3D) body is produced on film by irradiating the body with X-ray photons. Each ray is attenuated by a factor depending on the integral of the linear attenuation coefficient along the path of the ray, and produces a corresponding gray level at the point of incidence on the film. The radiograph so produced is a 2D projection (X-ray) of the 3D object, where the internal details are superimposed.

In X-ray computed tomography, a collimated X-ray beam passing through the desired cross-section of test object causes a line of projection on an array of detectors located on the other side of the object. The object is rotated and projections at a number of angular positions of the object are made. Measurements are made at a number of points along the line of projection, for each angular position. Measured projections are manipulated by a computer according to a reconstruction algorithm to produce a two-dimensional map of the X-ray attenuation coefficients in the irradiated cross-section. The principle of image reconstruction from projections is then used to compute the image of the section.

To image (reconstruct) a 3D body, a series of CT images of 2D sections are reconstructed (from the projections of corresponding 2D sections) to cover the complete body. This set of CT images can be manipulated in an image processor to obtain different views of the 3D body cross-sections across the body.

CT is not confined to X-ray alone. Other radiations that are used for making projections and 3D reconstructions are ultrasound (time of flight or attenuation), nuclear magnetic resonance (NMR), and nuclear emission (gamma rays, positrons, etc.). While physical parameter imaged may differ between these modalities, once the projection data are acquired, the reconstruction procedure is usually the same. Non-ionizing radiation techniques are of importance, and other forms of projections may be desired to image different physical parameters of the test object. Founded on a mathematical basis of projection reconstruction[8], this technique has gained analytical status.

CT is an ideal technique for a wide range of industrial research and production line applications. CT inspection systems offer significant advantages in process development, process monitoring, and quality assurance applications. Tomography of composites is finding extensive applications in aerospace industry. CT imaging techniques have also been successfully applied for imaging of live tree trunks, investigating two phase flows, and even in geophysical tomography. Other industrial applications where information provided by CT is invaluable are, inspection of hollow turbine blades, rocket motors, structural ceramics, etc. CT has the potential for offering analytical solutions for a variety of applications in nuclear field. Fuel sub-assemblies when subjected to high fluence of neutron and high temperature environment undergo dimensional and chemical changes of significance. These changes influence performance behaviour of fuel in a nuclear reactor. Neutron CT can give cross sectional information in a reliable manner. Gamma-ray emission tomography can be used to study the axial and radial distribution of fission products in nuclear fuel[9]. CT is used to find fissile material content in various pellets encapsulated in containers.

Eddy current modelling

A necessary prerequisite for the development of a model in eddy current testing (ECT) is to get an insight into the way in which the electromagnetic field/defect interactions take place in materials. This in turn would help in (a) predicting the impedance changes in the eddy current probes due to presence of defects in materials, (b) predicting the magnetic flux line contours and eddy current flow lines in materials at different test conditions like test frequency, lift-off and probe coil dimensions etc., and (c) simulating the test conditions which are in other words difficult to replicate in laboratory.

The differential equation governing the ECT phenomenon under sinusoidal steady-state conditions, is derived from the Maxwell's equations. This can be solved either by analytical or numerical methods. Analytical methods developed to date are not effective in handling odd geometries and the solution involves complex Bessel functions which are difficult to solve. On the other hand, numerical methods are effective in overcoming these problems.

A 2D finite element model has been developed at the author's laboratory to simulate the ECT phenomenon[2]. The model is most suited for axisymmetric geometries. The governing differential equation is solved using an energy-related function, which is a statement of energy balance in the solution region. Once the vector potential is found, any physically observable electromagnetic quantity such as eddy current density, flux density, probe coil impedance, etc. can be calculated or predicted. The impedance of a differential eddy current probe with similar coils wound in the opposite direction can be approximated by calculating and summing the impedance of each coil. Modelling studies have revealed that the finite element method (FEM) code has a number of potential uses for NDE development:

- as an 'experimental model' for simulation of electromagnetic NDT situations that are too difficult or expensive to replicate in a laboratory environment
- as an aid to the physical understanding of the interactions between electromagnetic fields and defects in the material/specimen under test
- as a design tool for the study of alternate probe geometries
- as a training mechanism for the development of automated defect characterization schemes.

This model has been successfully used for the optimization of an eddy current probe for locating garter springs, placed to maintain uniform gap between calandria tube and pressure tubes in Pressurised Heavy Water Reactors. The ECT probe thus designed with the aid of computer modelling has shown improved results during pre- and in-service inspection campaigns in various reactors, thus proving this approach to be an analytical NDE tool.

X-ray diffraction

Discovered in 1913 by Max Von Laue, X-ray diffraction is a method of analysis that utilizes the unique scattering of X-rays as a function of the crystal lattice structure. Some of the typical applications of this technique include determination of the crystal sizes, structure, composition, identification of phases, study of precipitation and age hardening and measurement of residual stresses. The technique can be considered to be qualitative, quantitative as well as analytical depending on the nature of the application. Thus, if it is used for the determination of phases, it could be considered to be qualitative while determining the composition of a given substance could classify this technique as quantitative. X-ray diffraction has been used since long as an analytical tool to study the crystal structure of materials. Now, X-ray diffraction techniques are being used to determine the strains, and in turn the stresses present in the material. It is well known that physical and chemical properties of a substance depend on the molecular and atomic arrangement. In this regard, X-ray diffraction is the viable NDE technique available for the measurement of interatomic spacing. While one can get a good idea of the interatomic spacing experimentally through this, theoretical modelling would help predicting the strains and stresses. This forms the basis of residual stress measurements by X-ray diffraction technique.

The application areas in the field of residual stress determination span from stress analysis of uranium fuel rods and aluminium alloy landing gear components to measurement of stresses in metal powder-doped polymeric materials, tempering evaluation of carburized steel and evaluation of abrasion damage produced in carbon graphite seal materials during service. While initially this technique was confined to the laboratory, the advent of solid state detector arrays has made it a field technique. A spatial resolution of about 0·1 mm is possible with the depth of penetration being about a few microns.

The recent advances in this field include the rotating anode microfocal X-ray generators which provide an increased intensity (about an order of magnitude) and resolution. This enables small areas to be examined for surface stress mapping and examining samples of awkward shapes and sizes. The other major development is the availability of synchroton radiation sources for diffraction applications. The major advantages of using synchrotons is that they provide highly collimated tunable source of radiation varying from 0.01 mm to 0.01 nm with intensities several orders of magnitude higher than in conventional machines (thus leading to reduced exposure times). The possibility of changing the wavelength results in the ability to vary penetration depths in a material, or to keep the Bragg angle constant and vary (hkl) to investigate anisotropy effects. The variation of penetration depth allows stress gradient with depth effects to be investigated.

Synthetic aperture focusing technique

The synthetic aperture focusing technique (SAFT) utilizes a large aperture focused probe, synthesized electronically. SAFT enables increasing the fundamental resolution and defect sizing accuracy. To explain the capabilities of SAFT, a wide angle compression probe and a point flaw in the specimen are assumed. When the transducer scans over the specimen, each reflected echo for various scan positions with respect to the position of closest approach of transducer to the flaw is delayed in time due to the greater distance travelled by ultrasonic waves. If the individual scans are shifted by an amount equal to their predicted time delays, they will come into coincidence with each other and when they are summed, the resultant will be a large amplitude response. If the same analytical procedure is repeated centered around another position (other than that of the closest approach), the above time shift compensation does not produce a set of self-coincidence scans which results in a significantly smaller response. The time shifts can be achieved either electronically or digitally using a computer[3]. This technique is an excellent example of the advantages that accrue from the combination of conventional and advanced techniques based on analytical approaches. This technique is finding extensive application in monitoring crack geometry in the structures which is of immense value in predicting structural integrity based on fracture mechanics concepts.

Split spectrum processing

This technique is implemented by splitting the frequency spectrum of the received signal by using gaussian overlapping band pass filters having central frequency at regular intervals[4]. For the N number of filters used, if we take inverse Fourier transform, we get N number of time domain signals. These N number of time domain signals are subjected to algorithms such as minimization and polarity thresholding for extracting useful information. Split spectrum processing technique is widely applied in the analysis of signals from noisy materials like centrifugally cast stainless steels, carbon epoxy composites, welded joints and cladded materials[4]. It must be mentioned that this NDE technique is based on established theoretical methodology.

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1. Sage, A. P., in *Int. Encyclopedia of Robotics* (ed. Dorf, R. C.), John-Wiley and Sons Inc. USA, 1988, vol. 1, pp. 293–298.
2. Rao, B. P. C., Shyamsunder, M. T., Bhattacharya, D. K. and Baldev Raj, *J. Nucl. Technol.*, 1990, **90**, 389–393.
3. Thomson, R. N., *Br. J. Non-Destr. Test.*, 1984, **26**(5), 281–290.
4. Rose, J. L., Karpur, P. and Newhouse, V. L., *Mater. Eval.*, 1988, **46**(1), 114–122.
5. Singh, G. P. and Udpa, S., *Non-Dest. Test. Int.*, 1986, **19**(3), 125–132.
6. Kumara, S. R. T., Joshi, S., Kashyap, R. L., Moodie, C. L. and Chang, T. C., *Int. J. Prod. Res.*, 1986, **24**(5), 1107–1125.
7. Griffiths, D. G. and Purohit, B. K., *Br. Telecom Technol. J.*, 1991, 9(3), 88–96.
8. Marr, R B., *J. Math. Anal. Appl.*, 1974, 357–374.
9. Levan, F. *et al.*, *Feasibility Studies of Computed Tomography in Partial Defect Detection of Spent BWR Fuel*, STUK-A97, Finnish Centre for Radiation and Nuclear Safety, Oct. 1990.

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Fission track age of zircon separates of tuffaceous mudstones of the Upper Siwalik subgroup of Jammu–Chandigarh sector of the Panjab Sub-Himalaya

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Fission track ages of zircon grains separated from tuffaceous mudstone/bentonitic tuffs of the Upper Siwalik subgroup of Chandigarh (Ghaggar section) and Jammu (Uttarbeni) are determined to be 2.14 ± 0.51 Ma and 1.59 ± 0.32 Ma respectively. The zircon grains are considered to be cogenetic with the ash falls, and hence provide age estimates of the eruptive event(s). Implications of these age estimates in the understanding of chronologic framework of the Upper Siwalik subgroup of the Jammu–Chandigarh sector of the Himalayan Foreland are briefly discussed.

TUFFACEOUS mudstones were reported from the Pinjor Formation of the Upper Siwalik subgroup in the area east of Chandigarh[1]. On the basis of the stratigraphic occurrence of the tuffaceous mudstones towards the base of the Pinjor Formation in the Ghaggar section, it was suggested[1] that these may correspond to the ash couplet dated between 2.4 ±0.20 Ma and 2.58 ±0.06 Ma in the Siwalik Group of Pakistan[2]. This surmise has now been investigated by fission track (FT) dating of zircon separates of the mudstones. FT ages of bentonitized tuffs from Nagrota Formation of Utterbeni area in Jammu Hills though reported in the literature are without any experimental and analytical details[3,4]. In view of this the FT age of zircon grains from bentonitic tuff bed from Utterbeni has also been attempted. Zircon was chosen for FT dating as it is fairly resistant to the process of thermal annealing of tracks. Present estimates place the long-term (10^6–10^7 years) annealing temperature of zircon at 225°–240° C (ref. 5). This value is sufficiently high for thermal events of lesser duration. The zircon grains are considered to be cogenetic with the ash fall(s) and should, therefore, date the eruptive event(s) and contemporaneous sedimentation. Further, burial of these tuffs to much less than 1 km for times of the order 10^6–10^7 years should have had no detectable fading effect on the spontaneous track densities of the zircon grains. They should, therefore, record a primary age.

Bulk sample (1–2 kg) was crushed and dispersed in water. Following wet sieving to remove clay minerals, the mineral concentrate was further sized to separate 60–200 mesh fraction. The zircon grains were separated using the conventional magnetic and heavy-liquid separation techniques.

FT dating was carried out at the Kurukshetra University Fission Track Dating Laboratory using external detector method in which the spontaneous fission tracks are etched and counted in the polished inner surface of the crystal and the induced fission tracks on an external muscovite detector (almost free from uranium) attached to the crystal during thermal neutron irradiation[6,7].

The euhedral and clear grains of zircon (considered to be cogenetic with a volcanic event) were mounted in PFA teflon. The mineral mount(s) were ground on a wet emery stone and polished with diamond pastes of 8 μm through 0.25 μm to obtain smooth surfaces with 4π geometry. The mounts were etched in an eutectic KOH–NaOH melt at 230°C for 26 h. In order to ensure optimum etching, the mounts were progressively etched till the appearance of fresh tracks stopped. The mineral mounts as well as two mounts of the standard dosimeter-glass CN1 (Corning-1) were covered with muscovite detectors and packed in an aluminium capsule. The capsule was irradiated in the IC2 thermal column of the CIRUS Reactor at Bhabha Atomic Research Centre, Bombay with a dose of $\sim 10^{15}$ n/cm^2. After irradiation, the muscovite detectors were etched in 48% HF at 30° C for 6 min to reveal the induced tracks.

The counting of tracks was done under Nikon optiphot microscope using 100 × dry objective. The grain surfaces of high etching efficiency recognized from the existence of sharp polishing scratches[8], were used for counting.

FT age was calculated using the zeta calibration approach. Experimental measurements of the zeta factor in the FT laboratory of Kurukshetra University for the dosimeter glass CN1 using the internationally accepted age standards and IC2 thermal column of CIRUS reactor has been determined to be equal to 110.56 ± 1.28 (1σ) (ref. 9). FT age was calculated using the following equation:

$$T = \frac{1}{\lambda_D} \ln \left[1 + \lambda_D \frac{G \zeta \rho_s \rho_d}{\rho_i} \right],$$

where λ_D is the total decay constant of uranium = 1.55125×10^{-10} yr^{-1} (ref. 10); ζ the zeta factor; ρ_s the spontaneous track density; ρ_i, ρ_d the induced track density in muscovite detectors attached to zircon sample and glass-dosimeter respectively; and G the geometry factor.

In order to estimate the errors, the data were subjected to χ^2-test[11] to detect the extra Poissonian error in track counts. The data failed the χ^2-test and hence instead of applying conventional analyses, the mean of the individual crystal track density ratios