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Nuclear Resonance for the Nondestructive Evaluation of Structural Materials

G. A. Matzkanin
Southwest Research Institute

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Abstract

It is appropriate that this presentation is in a session on new techniques because the program I will discuss this afternoon is sufficiently new that few concrete results are available to report. The program I will discuss was recently funded at Southwest Research Institute by AFOSR and involves examining the possibility of developing nuclear resonance techniques for the nondestructive evaluation of structural materials. What I hope to do this afternoon is to fill you in a bit with regard to the background involved in this program and bring you up to date regarding our research plans. I will discuss some of the experiments we hope to perform, and briefly describe a few results that were obtained in some related preliminary experiments.

Keywords

nondestructive testing, nondestructive evaluation

Disciplines

Materials Science and Engineering | Structures and Materials

NUCLEAR RESONANCE FOR THE NONDESTRUCTIVE
EVALUATION OF STRUCTURAL MATERIALS*

G. A. Matzkanin
Southwest Research Institute
San Antonio, Texas

It is appropriate that this presentation is in a session on new techniques because the program I will discuss this afternoon is sufficiently new that few concrete results are available to report. The program I will discuss was recently funded at Southwest Research Institute by AFOSR and involves examining the possibility of developing nuclear resonance techniques for the nondestructive evaluation of structural materials. What I hope to do this afternoon is to fill you in a bit with regard to the background involved in this program and bring you up to date regarding our research plans. I will discuss some of the experiments we hope to perform, and briefly describe a few results that were obtained in some related preliminary experiments. [1]

As I have said, the intention of this research program is to examine various NMR techniques with regard to their possibility for nondestructive evaluation of structural materials. The program will be primarily directed toward nonmagnetic materials, with interest initially centering on aluminum and aluminum alloys. It is intended that later in the program titanium and titanium alloys will also be investigated. In addition, we are expecting to look primarily at the residual stress problem in these materials. So, at least in the initial stage of the program we will be investigating the effects of internal strain and residual stress on NMR signals in aluminum specimens.

During the course of the program, we intend to pursue both conventional electromagnetic NMR approaches and acoustic NMR approaches. Participating in this program, especially with regard to the acoustic NMR aspects, is Professor Robert Leisure from Colorado State University. Besides utilizing his expertise in acoustic NMR, I believe having Professor Leisure involved in this program is important from the standpoint of increasing the participation of the university community in NDE research activities. Professor Leisure has been involved in recent years in a variety of acoustic nuclear resonance studies and is expected to contribute in this general area to this research program.

I don't want to belabor the fundamentals of NMR because most of you, I am sure, are familiar with nuclear magnetic resonance, but it might be worthwhile to take a few minutes to remind those of you who have not been exposed to this area for awhile just what is involved. In nuclear resonance we are interested in the resonant excitation of nuclear magnetic dipole moments. Figure 1 shows a semi-classical picture of a precessing magnetic moment in an applied magnetic field, H_0, where the precessional frequency, called the Larmor frequency, is proportional to the magnetic field. For most materials of interest, these precessional frequencies are in the neighborhood of 10 MHz for magnetic fields of 10 kilogauss. One achieves nuclear resonance by introducing radio frequency energy into the system at the appropriate frequency, namely, the Larmor frequency, so that there is a resonant interaction that takes place with the nuclear spin system. [2]

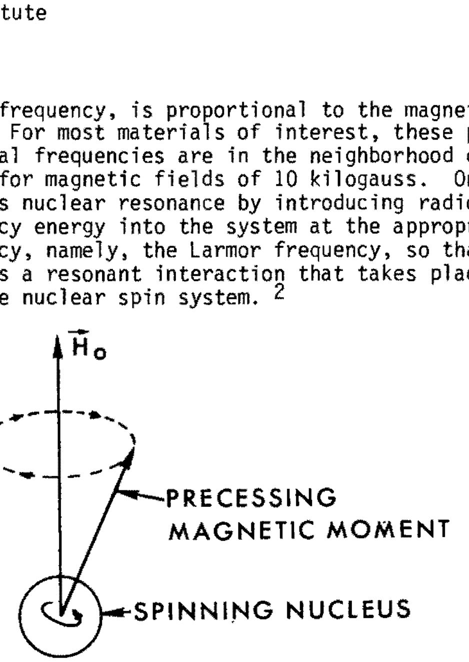

Figure 1 Precession of a Nuclear Magnetic Moment in a Static Magnetic Field.

In Fig. 2 are shown two basic experimental approaches that can be utilized for NMR, namely, the electromagnetic NMR approach whereby the energy is introduced into the specimen by means of an inductive coil which encircles the specimen, and the acoustic approach whereby the rf energy is introduced into the specimen by means of a transducer mechanically coupled to the specimen. By several possible mechanisms, the acoustic energy can couple to the nuclear spin system, and if at the appropriate frequency, can bring about a resonant interaction. [3]

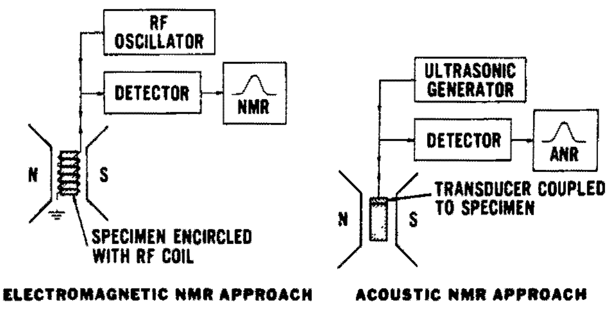

Figure 2 Experimental Methods for Nuclear Magnetic Resonance.

The electromagnetic NMR approach, while it is older and much better understood than the acoustic approach, has a fundamental limitation with regard to metals. That is, the radiofrequency fields can

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only penetrate into the skin depth of the metal and at the frequencies normally employed, this is nominally 10 to 100 microns. So from the practical standpoint of nondestructive evaluation of metals and alloys, the electromagnetic NMR approach is limited to the surface. The skin depth problem in metals can be circumvented by the acoustic NMR approach in which the energy penetrates into the bulk of the metal. In our research program, we are intending to investigate both methods. Although the electromagnetic method is limited to the skin depth in metals, it is much better understood than the acoustic method and there are theoretical models available for the effects of strain and internal stress that can be readily applied to the results. Also from the practical standpoint, there are many instances where surface residual stresses are of interest.[4]

Figure 3 is a schematic diagram of the nuclear magnetic resonance process. Energy, either acoustically or inductively, is introduced into the nuclear spin system. If the corresponding frequency is appropriate to cause transitions between the magnetic energy levels of the system, a resonant interaction takes place and energy is absorbed by the nuclei. If the applied magnetic field, H, is varied, one observes a typical bell-shaped signal. As we shall see in a moment, it is in the characteristics of this signal where the information regarding residual stress and strain lies. So the investigation centers around a study of the effect of stress and strain on the various parameters of this signal, such as amplitude, width, shape, and frequency.

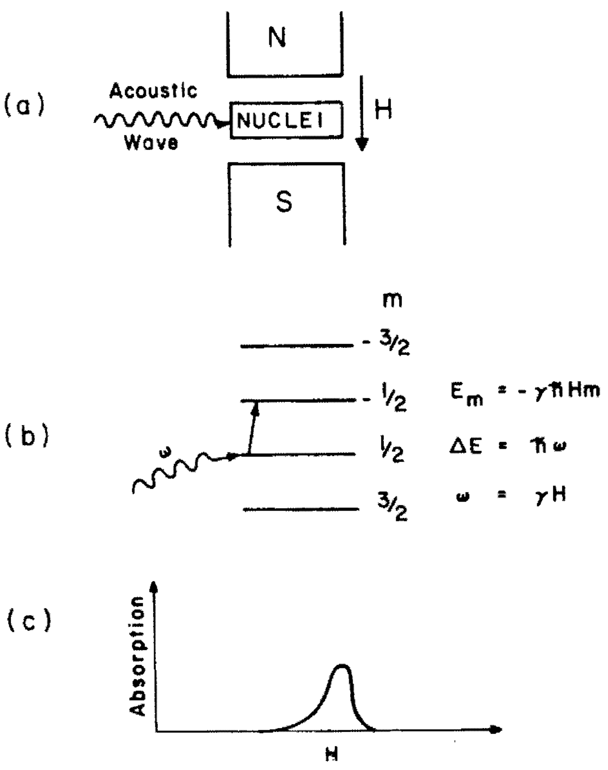

Figure 3. Schematic Diagram of a Nuclear Magnetic Resonance

The dominant physical mechanism by which residual stress and strain enter the picture, is the nuclear quadrupole interaction, shown schematically in Fig. 4. Many nuclei, in addition to a magnetic moment, also possess a nuclear quadrupole gradient set up by other ions and electrons in the material. It is by means of this interaction that information regarding stress and strain is manifested in the NMR signal. In general, the quadrupole interaction vanishes for cubic symmetry. However, even in cubic crystals, several perturbations may exist so that electric field gradients are produced. Three of these possibilities are listed in Fig. 4: (1) stress/strain fields produced by external loads or lattice defects; (2) charge difference between point defects and the host ions; and (3) redistribution of conduction electrons around a defect in the case of metals.

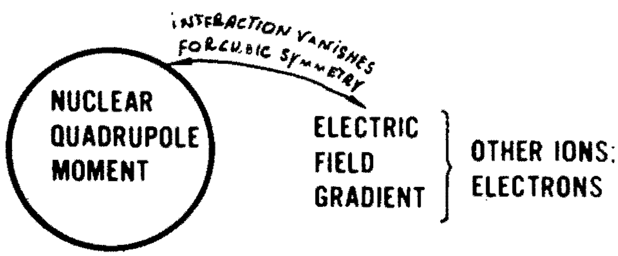

Figure 4. Schematic Illustration of Nuclear Quadrupole Interaction

Figure 5 illustrates schematically how the energy levels for a magnetic system are perturbed by the presence of an electric field gradient. The case illustrated is for a nuclear spin of five-halves which would be the case appropriate to aluminum. For a magnetic field only, the energy levels are all equidistant; however, with a quadrupole interaction present, the energy levels are shifted so that the NMR signal which would be observed in the case of no quadrupole effects is affected in various ways depending on the circumstances. The result is that the quadrupolar perturbations that are present generally have an observable effect on the various parameters of the net nuclear resonant signal.

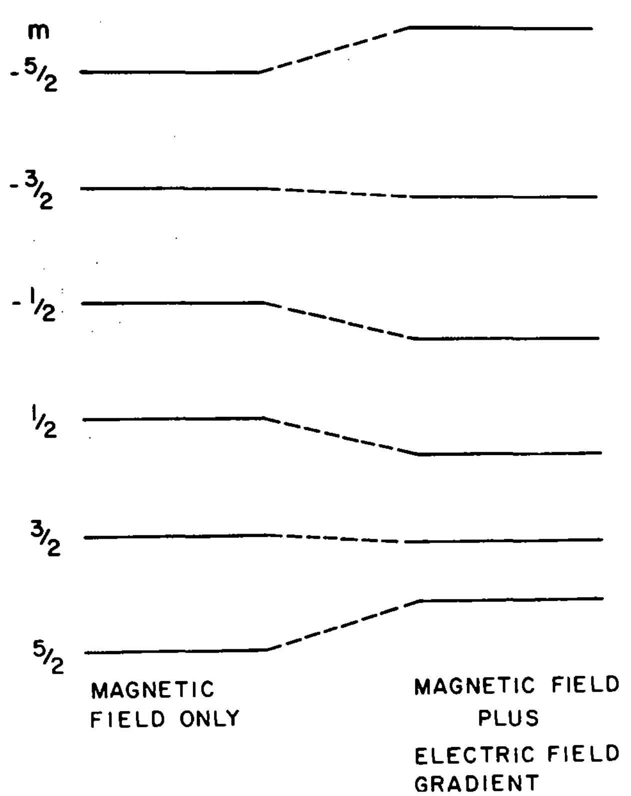

Figure 5 Energy Level Diagram of a Spin $^5/2$ System, Showing Electric Quadrupole Effects.

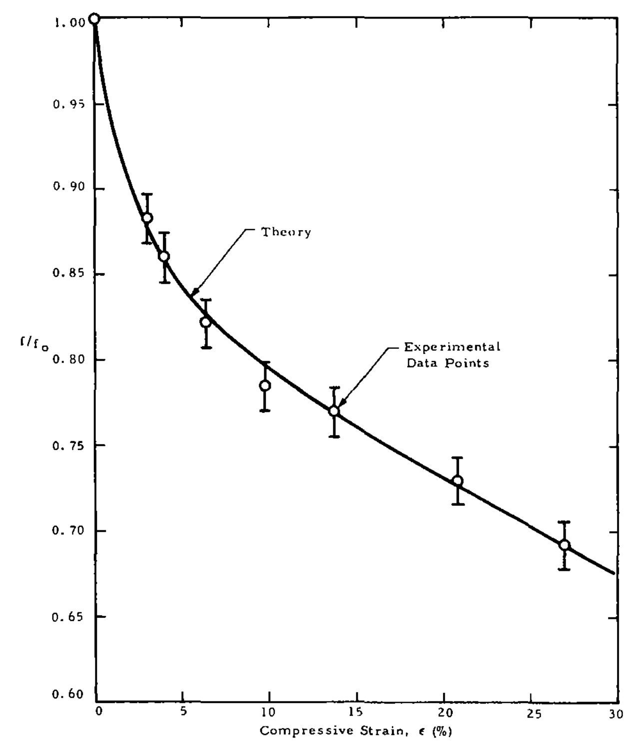

Figure 6. ^{27}Al Normalized Peak-to-Peak NMR Signal Intensity in Bulk Polycrystalline Aluminum versus Compressive Strain

Some theoretical work has been performed by Kanert and his co-workers in Germany where they have calculated the electric field gradients due to various kinds of defects. Specifically, they have calculated the electric field gradients for point defects including both strain and valence effects; for dislocations, both screw and edge types; and for dislocation dipoles and tilt boundaries. They calculated the electric field gradient appropriate to these various kinds of defects and analyzed the way in which these electric field gradients affect a nuclear magnetic resonance signal for the case of cubic symmetry. [5]

We have performed some experiments to investigate these theoretical approaches. Figure 6 shows some of the results obtained on bulk aluminum specimens. These experiments were performed with the conventional electromagnetic NMR techniques where, as was indicated earlier, only the skin depth layer of the bulk metal specimens is sensed. The specimens were polycrystalline aluminum cylinders that were deformed in compression to various amounts. The continuous wave approach was utilized and the intensity variation in the signal was compared to the theoretical analysis provided by Kanert's approach. As is seen in Fig. 6 the agreement is quite good. These data can be examined in another way whereby the dislocation density is obtained from the measured changes in the NMR signal intensity using the theoretical development of Kanert. These results can be plotted against the flow stress for this specimen, and, as is shown in Fig. 7, the expected linear relationship is obtained.

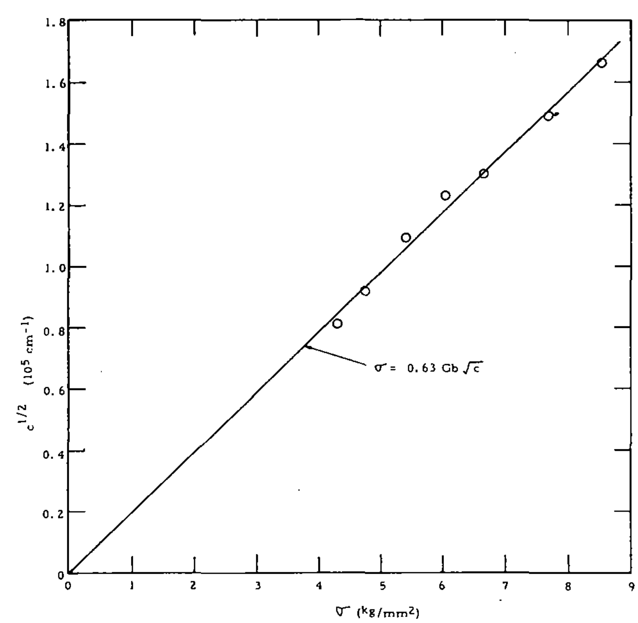

Figure 7. Square Root of the Mean Dislocation Density c in Polycrystalline Aluminum as Determined from NMR Signal Intensity Measurements versus Compressive Stress

As far as acoustic nuclear resonance goes, no studies of the effects of strain have been performed yet, but some recent work by Prof. Leisure and his co-workers illustrates acoustic NMR signals that have been observed from aluminum.[6] In Fig. 8, at the top, is shown an acoustic nuclear resonance absorption signal which was observed in a single crystal of aluminum. At the bottom of Fig. 8 is shown an acoustic NMR signal which was obtained in an aluminum-zinc alloy. The specimen was a dilute alloy of about 1 percent zinc. The observed signal was a dispersion signal, as opposed to an absorption signal, and is associated with the change in the acoustic velocity.

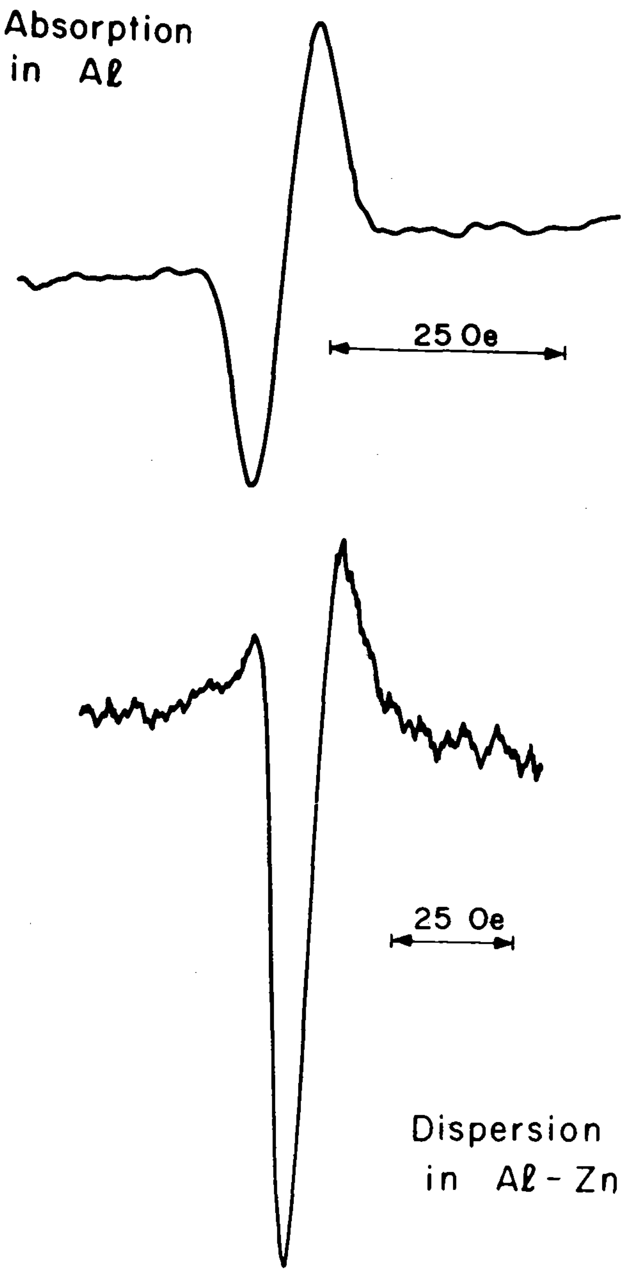

Figure 8. Representative Acoustic Nuclear Resonance Signals

Prof. Leisure has also investigated the satellite signals that appear in dilute alloys by the acoustic nuclear resonance approach. In the case of a material like aluminum, which is normally a cubic crystal structure, if an impurity such as zinc is introduced, then the aluminum atoms which are close to the zinc nuclei will feel the effects of the quadrupolar interactions and the electric field gradients which are produced in the specimen by the presence of the zinc impurities. Thus, those aluminum atoms which are close to zinc atoms will have a resonance signal separated from the main resonance which is observed for the majority of aluminum nuclei not near zinc atoms. This gives rise to the so-called satellite signals as shown in Fig. 9 (only one side of the signal is shown). These satellite signals can be associated with aluminum atoms which are surrounding the zinc atoms -- first neighbor, second neighbor, or whatever the case may be -- and thus affords a mechanism for studying the quadrupole effects introduced by the zinc atoms.

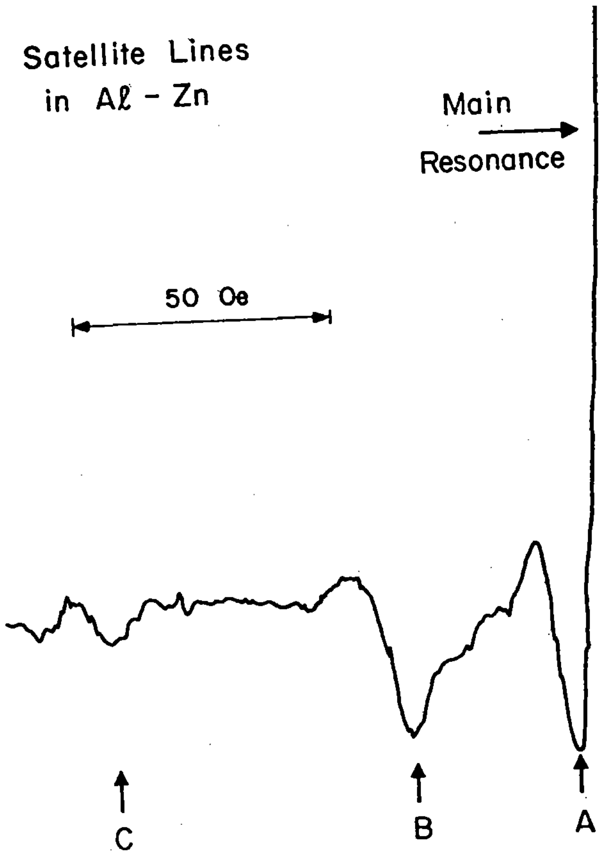

Figure 9. Acoustic Nuclear Resonance Signal from an Aluminum-Zinc Alloy

Although it will take much work to realize the potential of NMR for nondestructive evaluation, a summary of what we are hoping to accomplish in this particular program is shown in Fig. 10. The first thing we hope to do is to investigate the effects of strain using encircling coils and homogeneous fields. These will be relatively straightforward conventional NMR experiments where we will take specimens of aluminum and, using the usual encircling coil techniques, we will investigate specimens which have been strained in various ways with both applied and residual stress. We will compare the results of these experiments to the theoretical results that are available primarily from the work of Kanert and his co-workers. Within the context of this task, we will investigate the effects of strain fields on the parameters that one measures in a nuclear resonance experiment. We expect to use primarily pulsed NMR approaches where the relaxation times and nuclear spin echoes can be interpreted in terms of the electric field gradients which exist in the specimen and thus be related to strain fields.

TASKS

Task 1. Investigation of Effects of Strain Using Encircling Coils and Homogeneous Fields

Task 2. Development of Non-encircling Coil Techniques and One-Sided Magnet Techniques

Task 3. Preliminary Investigation of Effects of Lattice Strain by the ANR Technique

Task 4. Extension of Non-encircling Coil and One-Sided Magnet Techniques to Lattice Strain Measurements

Task 5. Extension of ANR Techniques

Task 6. Parametric Study for Prototype Practical Instrumentation

Figure 10. Outline of Program to Investigate and Develop NMR Methods for the Nondestructive Evaluation of Residual Stress in Structural Materials

Task 2 involves an investigation of nonencircling coil and one-sided magnet techniques. Eventually, of course, we are interested in practical application of these techniques, and therefore, one would like to be able to perform NMR experiments using nonencircling coils, where the specimen is removed from the coil, and also using magnetic fields that are somewhat less homogeneous than those available in the laboratory. So within the context of Task 2, we expect to begin looking at these possibilities. We have already done some work along these lines in another program underway at our laboratory. Although the situation is somewhat simpler, namely, detecting proton NMR signals, the success achieved using pancake-shaped rf coils and one-sided magnets indicates that these approaches are, at least, feasible.

Task 3 involves an investigation of the effects of strain by the acoustic NMR technique. Here, we will essentially be extending Task No. 2 in that some of the work performed in that task will be applied to using acoustic NMR to investigate signals obtained from strained specimens. In this task, we will initially work with relatively well understood specimens such as alkalide-halides. There has been a good deal of acoustic NMR work performed on these materials so they present a good starting point for investigating strain effects. After understanding the effect of strain on acoustic NMR in alkalide-halides, we will extend the investigation to aluminum.

Task 4 involves investigations of extensions of acoustic NMR techniques. There are several things that it would be useful to pursue with regard to the potential application of acoustic NMR for NDE purposes. Some of these we expect to pursue in this task. For example, the acoustic NMR work that has been performed so far has been on single-crystal materials. Also, we plan to pursue the possibility of utilizing pulsed techniques in acoustic NMR.

Task 5 involves incorporation of nonencircling coil and one-sided magnet techniques into the work that was done in Task 1 and 2, namely, the investigation of strain.

Finally, Task 6, a little further down the line, involves what will eventually be needed in order to bring this work to practical realization. In this task we would hope to perform a parametric study of the parameters that are needed in order to breadboard stress-measuring instrumentation for practical implementation.

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DISCUSSION

PROF. JOHN TIEN (Columbia University): Thank you. We have time for a couple of questions before coffee.

DR. BRUCE MAXFIELD (Livermore Labs): Were the line widths that you showed typical line widths of the order of 10 to 15 kHz?

DR. MATZKANIN: Do you mean the acoustic NMR line widths?

DR. MAXFIELD: Yes.

DR. MATZKANIN: Yes. For aluminum that would be a typical line width. The specimen was a crystal, by the way.

PROF. TIEN: Would you put some time frame on those tasks? Are they a month apiece?

DR. MATZKANIN: I was afraid you would ask that. We are thinking in terms of about a three-year program for what you see there, for those six tasks. That, of course, is assuming that everything goes as planned and things fall together as we would like.

PROF. TIEN: Also, you keep mentioning aluminum,and I hope I didn't hear right, alkali-halides?

DR. MATZKANIN: That's right. Do you mean why did I say that?

PROF. TIEN: No. Does this NMR not like titanium or anything else?

DR. MATZKANIN: That's a good question. Titanium, of course, is something that we are interested in and, yes, you can perform nuclear resonance on titanium and it has been done. However, there are some difficulties involved, primarily the fact that the NMR frequency in titanium is very low and somewhat weaker than aluminum. For example, in a magnetic field of 10 kilogauss, the resonance frequency for titanium is only about 2 MHz. So it is very low, and it is a difficult signal to observe. There may be ways to get around this, for example, by using signal averaging techniques and pulsed approaches. It's not out of the picture. We hope to look at titanium within the context of the program.

PROF. TIEN: How about things like nickel and higher temperature alloys? Are there any material restrictions?

DR. MATZKANIN: One of the slides I didn't get to since we ran out of time listed other things that one might think of doing within the context of nuclear resonance, and one of these is ferromagnetic resonance. This is an approach that one can use in magnetic materials where the internal magnetic fields are set up internally to the materials. It would certainly be of interest to pursue these investigations for application to steels, nickel and so forth. NMR experiments have been performed on these materials; however, we have not included them within the context of this program since we have to limit the scope.

PROF. TIEN: All right, one more question.

DR. R. CLOUGH (National Bureau of Standards): Would you care to make any speculations about using X-ray or neutron excitation?

DR. MATZKANIN: In what way now? I'm not sure I understand --

DR. CLOUGH: Instead of phonons.

DR. MATZKANIN: Instead of phonons? I don't know. As far as coupling to the nuclear spin system goes, I'm not sure what mechanisms would be available to do this. I don't know of any work in that area currently going on.

PROF. TIEN: Thank you.