JOURNAL OF MATERIALS SCIENCE **39** (2004) 6555–6559

Free nitrogen effect on creep failure and creep crack growth of C-Mn steel

MOON SIK HAN*
Faculty of Mechanical and Automotive Engineering, Keimyung University, 1000 Sindang-Dong, Dalseo-gu, Daegu 704-701, Korea
E-mail: sheffhan@kmu.ac.kr

OH GEON KWON
Materials Performance Technologies, Industrial Research Limited, PO Box 31-310, Lower Hutt, Wellington, New Zealand

Different chemical compositions and heat-treatments of the C-Mn steels were investigated to characterise a susceptibility to cracking on cold bent tube. The C-Mn steels were categorised in terms of the content of free nitrogen. Ultimate tensile strengths, yield strengths and elongations were measured from the tensile testing at ambient, 250 and 360°C. Significant increase in ultimate tensile strength and decrease in elongation were observed in high free nitrogen material at 250°C the temperature at which free nitrogen is most active. From the uniaxial creep testing on high free nitrogen materials at 360°C increase in creep property, for example, lower minimum creep rate, was observed, however there was a decrease in creep ductility. This lower ductility of the high free nitrogen material has provided higher susceptibility to cracking in the creep crack growth tests at 360°C. Cracking in the high free nitrogen material A was approximately three times faster than the low free nitrogen material C at the same C* value. The creep cracking and rupture life in the high free nitrogen materials were more sensitive to the material condition, for example, pre-straining and/or heat treatment because of the role of the free nitrogen in the steels.

1. Introduction

Over the last 30 years failures in cold-formed bends in power station boiler manufactured from Carbon Manganese steel (C-Mn steel) have been regularly reported. The failures in many cases initiated longitudinally at the pipe outside surface through a slow crack growth mechanism followed by fast fracture. The pipe and bends in the boiler system generally operated at approximately 360°C and 168 bar.

At these operating conditions, potential crack growth mechanisms are regarded as fatigue, stress corrosion and creep. However, it has been identified that creep crack growth is the most likely mechanism of cracking in the relatively low temperature of operation of the pipework system [1].

Earlier investigations carried out on boiler pipes and tubes which failed in service through creep crack growth identified that the level of free nitrogen (FN) might have significant influence on the creep crack growth characteristics and service performance of carbon-manganese steels operating between 300–420°C. It was found that there was a considerable variation in susceptibility to failure in steels made to the same specification. A high free nitrogen content was identified as a common factor among the components susceptible to failure [2–4]. It has been established by Hopkin that obstacles to dislocation mobility were formed by the clustering of manganese and nitrogen atoms in solid solution [5]. These clusters impede dislocation movement and affect the rates of work hardening and recovery during creep. Thus the creep resistance of the carbon steel may be enhanced by increasing the level of the free nitrogen content, however a susceptibility to cracking may be increased.

In this work the effect of free nitrogen on tensile properties, uniaxial creep properties and creep crack growth behaviour of C-Mn steel with a range of free nitrogen content is considered. In addition other factors, affecting susceptibility to creep cracking and failure, such as pre-strain due to cold bending and heat treatment are also discussed.

2. Materials

The chemical composition of each batch of C-Mn steel is shown in Table I. They are designated A to F.

Three methods were used for the determination of free nitrogen contents, by calculation using N and Al

*Author to whom all correspondence should be addressed.

TABLE I Chemical analyses of C-Mn steel (wt%)

	C	Si	Mn	P	S	Cr	Mo	Ni	Cu	Al(tot)	N	FN†
A	0.16	0.28	0.87	0.014	0.014	0.09	0.05	0.2	0.15	0.019	0.019	0.011
B	0.17	0.24	0.87	0.015	0.015	0.09	0.05	0.2	0.15	0.005	0.017	0.0101
C	0.16	0.27	0.89	0.013	0.015	0.090	0.05	0.2	0.15	0.043	0.015	0.0013
D	0.15	0.25	1.08	0.008	0.011	0.14	0.04	0.13	0.18	0.016	0.018	0.0111
E	0.16	0.18	0.57	0.006	0.023	0.19	0.08	0.2	0.24	0.015	0.011	0.0087
F	0.14	0.2	0.75	0.007	0.007	0.15	0.05	0.18	0.14	0.02	0.014	0.0037

A: High FN plate, B: Medium FN plate, C: Low FN plate, D: Susceptible tube, E: Susceptible pipe, F: Non-susceptible pipe, †FN is calculated "Free nitrogen" for 920°C for all materials.

TABLE II Ranking of materials in terms of free nitrogen contents determined by different methods

'Al-N' method		'Thermocalc' method		Internal friction method	
A, D, E	High	D	High	A, F	High
B	Medium	B, F	Medium-High	B, D, E	Medium
F	Medium-Low				
C, G	Low	C, E	Low	C, G	Low

contents only, by the use of a 'Thermocalc' programme which takes account of nitrogen fixing elements other than Al, and direct measurement from an internal friction method using a torsion pendulum [6]. Ranking as high, medium and low was made rather than deciding a specific value of the free nitrogen content for each material due to the difficulties in obtaining a definitive free nitrogen content on the basis of analysis results from a single laboratory and method. A, D were grouped as high level of free nitrogen materials and B, E and F as medium and C as low free nitrogen materials.

The ranked material based on the analysis results from the three different methods are shown in Table II. In spite of the variation of ranking among the three methods, A, D can be grouped as high level of free nitrogen materials and B, E and F as medium and C as low free nitrogen materials.

The corresponding mechanical properties of batches A to F at three different temperatures are illustrated in Fig. 1.

It is clear from Fig. 1 that there is an expected tendency of decrease in yield strength as temperature is elevated. This can be understood by considering deformation as a thermally activated process. At low temperature a high stress to achieve a given strain rate is needed, on the contrary at high temperature, where more thermal energy is available, a lower stress is necessary [7]. The ultimate tensile strength (UTS) generally decreases with temperature, however the UTS in most batches except C material, which is regarded as low free nitrogen material, increases with temperature, because the presence of free nitrogen hinders dislocation movement. It is also found that the UTS in high and medium free nitrogen materials reach a peak at around 250°C (the temperature at which free nitrogen is most active).

The difference between the ultimate tensile stress (UTS) at ambient and 250°C is shown in the form of a bar-chart in Fig. 2. It can be seen that the high and medium FN materials show positive and significant ΔUTS values. On the contrary, the low FN material C shows a negative value. This indicates that the determination of the ΔUTS value may be an indicator of free nitrogen content in the steel. Fig. 2 shows the biggest value of the ΔUTS was observed at the F material which was originally purchased as medium low FN material based on chemical analysis.

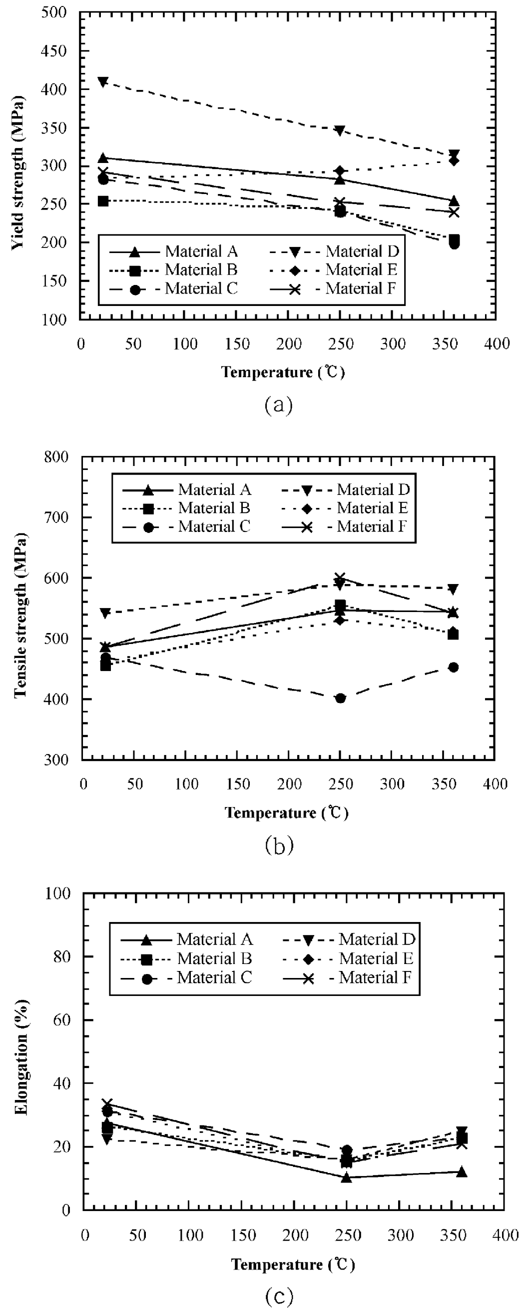

Figure 1 Mechanical properties of C-Mn steel at three different temperatures: (a) Yield strength of C-Mn steel at three different temperature, (b) Tensile strength of C-Mn steel at three different temperatures, and (c) Elongation of C-Mn steel at three different temperatures.

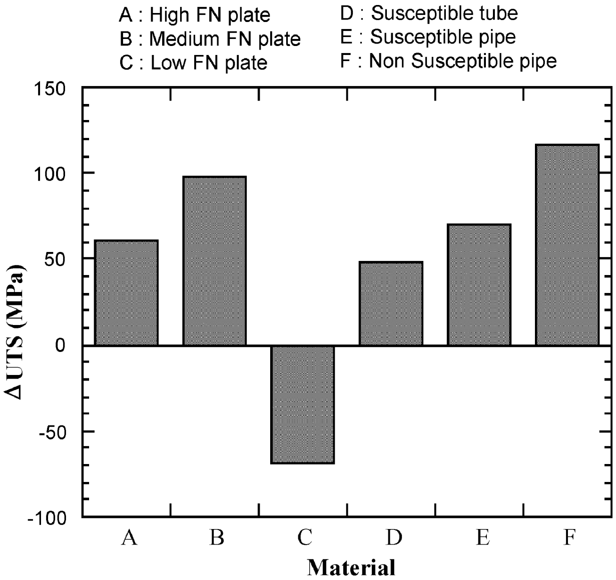

Figure 2 Difference of Ultimate Tensile Stress(UTS) between ambient and 250°C.

3. Creep

Uniaxial creep rupture tests on plain bar specimens of 5.64 mm diameter for A to F material were carried out at 360°C. Creep failure strain and reduction of area of the fractured specimen were measured. The minimum creep rate was evaluated as a representative creep strain rate. The average creep rate which accounts for all stages of creep in an approximate way was also obtained from creep failure strain and rupture life and compared with the minimum creep rate.

Normalised creep curves of the different materials A to F at 360°C are shown in Fig. 3 to compare the shape of the creep curves. Primary, secondary and tertiary creeps are clearly seen in Fig. 3. The creep curves of the high and medium FN material A, B, D and E were distinguished from that of the low FN material C which had bigger primary and secondary.

The minimum creep strain rates against stress of Material A to F at 360°C are shown in Fig. 4. Approximately the same slope $n \approx 18$ in the Norton creep law can be drawn between each set of data. Material A

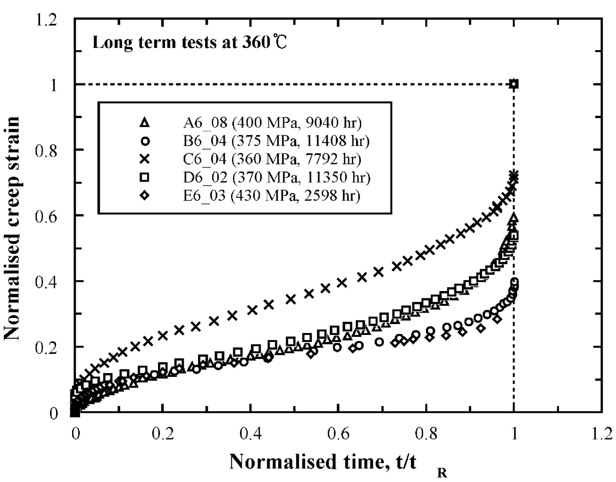

Figure 3 Normalised creep curves for long term tests of the materials at 360°C.

TABLE III Creep properties of material A to F at 360°C

	Material A, B, D, E, F	Material C
Minimum creep rate		
A	4.4E-53	1.17E51
n	18.2	18.2
Ductility (average)%	18	30
v	14	14

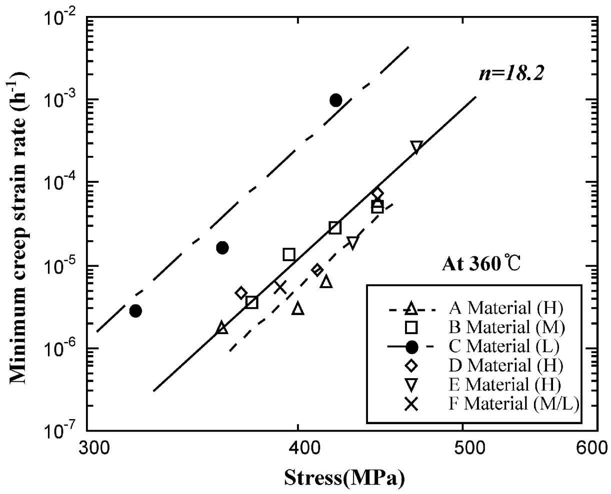

Figure 4 Minimum creep strain rate against stress of Material A to F at 360°C.

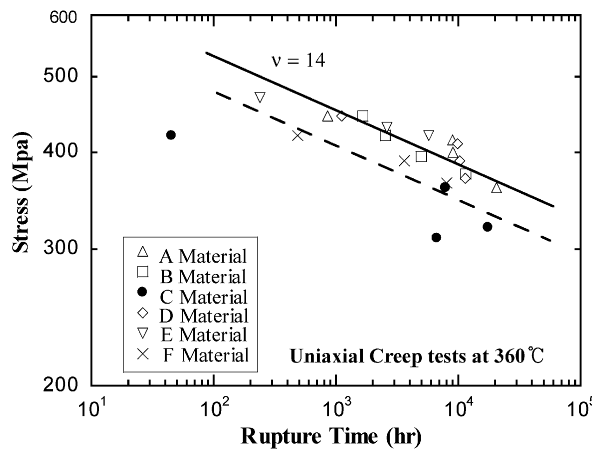

Figure 5 Uniaxial rupture data for all Material at 360°C.

shows slightly the slowest creep rate, with little difference between material B, D, E and F. Material C with the lowest free nitrogen content is clearly the weakest.

The uniaxial creep rupture data of all materials at 360°C are expressed by the relationship between net section stress and rupture life in Fig. 5. It can be seen that a linear relationship with the same slope ($\nu = 14$) is found. Fig. 5 shows that the weakest creep rupture behaviour appears in the low free nitrogen material C.

The material properties for characterising creep behaviour of material A to F are shown in Table III.

4. Creep crack growth

Creep crack growth (CCG) tests were conducted on pre-strained and heat treated compact tension (CT) specimens machined from the high and low free nitrogen (FN) materials A and C at 360°C. The pre-strained CT specimens were manufactured from large tensile which were pre-strained to 15% (the axial strain on a typical pipe bend extrados) in order to study the effect of cold straining on CCG characteristics of both high and low FN steels. Some CT specimens were heat-treated for 3 h at 650°C after 15% of pre-straining to study the use of stress relief heat-treatment as a cure for in-service bends. Some of the heat-treated CT specimen were renormalised.

Constant load tests at two specific loads were carried out using lever type creep machines with a lever ratio of 10:1. The AC PD (potential drop) system was used as a means of measuring crack length. In order to measure the temperature of a specimen thermocouples were also spot-welded either side of the crack plane. The temperature was controlled and monitored by the thermocouples. The temperatures were kept within ±1°C from the setting temperature. Load line displacement was continuously measured using a LVDT and recorded on a chart recorder and a computer. During and after application of the required load, the displacement transducer readings were recorded continuously throughout. Initial values of the crack length and the PD voltage were also recorded and the subsequent values were collected on chart recorders and a computer data acquisition system.

Figs 6 and 7 show correlations of CCG rate with C* for pre-strained, heat-treated and renormalised CT specimens made from materials A and C. These characteristics of CCG behaviour of material treated specimen made of high FN material A were compared with those from as-received specimen as shown in Fig. 6. It is clear that the pre-strained material fails at a faster rate. It is argued that this is because of the decrease in creep ductility due to the pre-straining to 15% strain which caused an increase in cracking rate of a factor of about 2. When the pre-strained material is heat-treated at 650°C for 3 h, the free nitrogen is precipitated as silicon nitrides, in other words the free nitrogen content is reduced. Then the density of dislocation will be decreased with increased ductility. Hence the effect of heat treatment on pre-strained material recovers its creep cracking properties. When renormalising treatment was applied to this material, it provided the lowest cracking rate, although this treatment was intended to recover its original properties. It is evident that renormalisation resulted in a significant increase in creep ductility of the material. However this could not be checked as the creep deformation properties in this condition were not measured.

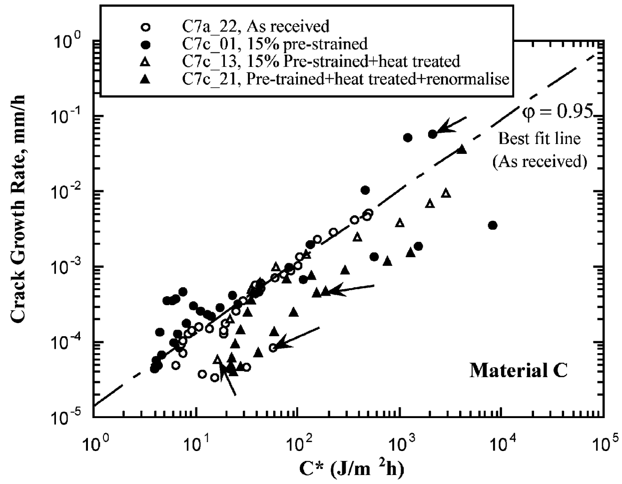

Figure 7 Effect of material treatment on CCG rate for Material C at 360°C (arrows indicate starting point).

In Fig. 7 the effects of pre-straining and heat treatment are shown for material C. Similar trends are observed to material A, although the influence of subsequent heat treatment after pre-straining is less predominant.

The effect of several material treatments on the failure times of the creep crack growth tests for materials A and C was examined in Figs 8 and 9. For material A, the application of 15% cold work results in a very significant decrease in life for a given reference stress. When this material is then heat treated at 650°C

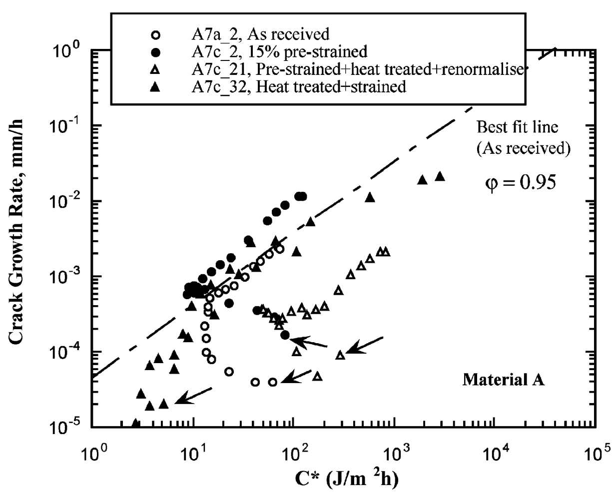

Figure 6 Effect of material treatment on CCG rate for Material A at 360°C (arrows indicate starting point).

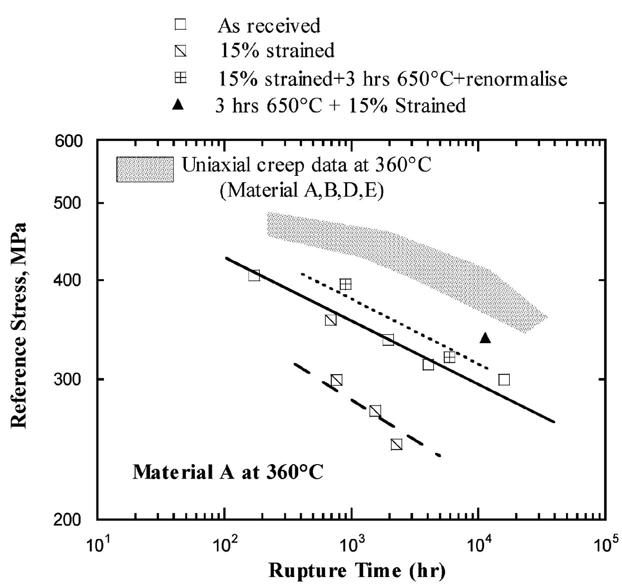

Figure 8 Material treatment effect on creep failure behaviour for CCG tests of Material A at 360°C.

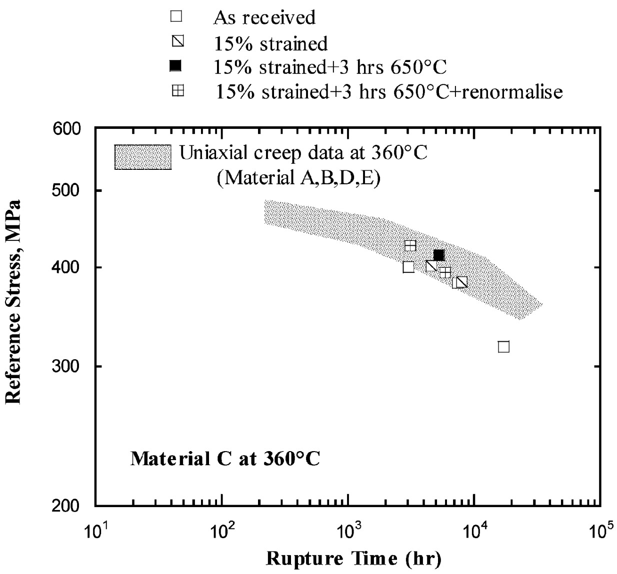

Figure 9 Material treatment effect on creep failure behaviour for CCG tests of Material C at 360°C.

for 3 h, lifetime is increased, not only back to that of the as received condition, but substantially longer than this.

The reason for this may be due to the precipitation out of free nitrogen as silicon nitride when the heat treatment was introduced to the material. Renormalising appears to return the behaviour to that of the as received condition. Finally, if material A is heat treated first, and then strained, the treatment leads an increase in life over the as-received condition. This possibly indicates that the heat treatment has a large and beneficial effect on the creep crack growth behaviour, whether the material is cold strained before or after this heat treatment. It suggests that application of this heat treatment may be used to reduce the risks of cracking in susceptible C-Mn steel components.

In Fig. 9, the effect of several material treatments on creep failure behaviour of the CCG tests is shown for Material C. In this case there is no difference in the creep crack growth failure times of as-received, prestrained and heat-treated material. The reason for this is possibly due to low content of free nitrogen in this material. Renormalising again appears to restore the original properties.

These results can be summarised that straining or cold work on high FN material deteriorate its CCG resistance and heat treatment on high FN material improve its CCG resistance, but not on low FN material.

5. Conclusions

The C-Mn steels have been categorised in terms of the content of free nitrogen. For high free nitrogen material higher ultimate tensile strength and lower ductility were observed than for low free nitrogen material.

The difference between the ultimate tensile strength at ambient and 250°C was determined for the materials A to F. The high free nitrogen materials showed positive ΔUTS values and the low free nitrogen material showed negative value. The results indicated that the determination of the ΔUTS values may be an indicator of free nitrogen content in the C-Mn steel.

The lower ductility of the high free nitrogen material has provided higher susceptibility to cracking in the presence of sharp notches or cracks. Materials A, B, D, E, F had similar creep properties, and C was weakest but had highest ductility.

For the high free nitrogen material, the creep cracking property was greatly affected by prestraining and heat treatment because of the role of the free nitrogen in the material. On the contrary, the low free nitrogen material was less sensitive to pre-strain and heat treatment than the high free nitrogen material. It can be expected from this fact that pre and post heat treatment should be effective in reducing the risk of cracking in C-Mn steel pipe.

References

1. D. J. GOOCH, *Mater. Sci. Engng.* **64** (1984) 183.
2. I. A. SHIBLI, *Mater. Sci. Tech.* **3** (1987) 110.
3. G. J. NEATE, *ibid.* **3** (1987) 14.
4. C. RINALDI, E. LUCON and F. CASAROLI, *Fatigue Fract. Engng. Mater. Struct.* **20**(8) (1997) 1159.
5. L. M. HOPKIN, *J. Iron Steel Inst., London* **203** (1965) 583.
6. R. V. MASKELL, "Creep Crack Growth in Carbon-Manganese Steels at 300–420°C", 2nd Annual Progress Report, BRITE/EURAM 7463, Mitsui Babcock Energy Limited 1996.
7. C. R. BARRETT and A. S. TETELMAN, "The Principles of Engineering Materials" (Prentice-Hall, New Jersey, 1973).

Received 29 May 2003
and accepted 30 June 2004