

Most metals and alloys have a negative temperature coefficient of modulus of elasticity; that is to say they lose stiffness when heated. They also have a positive coefficient of thermal expansion, increasing in length when heated. These two effects are due to increase in energy of the atoms with increase in temperature. Some ferromagnetic materials, however, exhibit markedly different behavior, which can be utilized to design constant modulus alloys.

The modulus of elasticity (E) of ferromagnetic materials is a complex function of a number of physical properties, related by the adjacent equation.

Each of the factors λ , μ and k is affected by composition, strain and temperature. In order that the modulus of elasticity will remain constant with variation in temperature it is necessary to select a

composition for which $\lambda^2\mu$ changes at the same rate and in the same direction as k^2 .

The same constants are also sensitive to applied magnetic fields so that the modulus of elasticity changes with a change in magnetic field strength.

As these considerations apply only to ferromagnetic materials they do not hold when an alloy has been heated beyond its Curie temperature, the point at which it changes from ferromagnetic to paramagnetic behavior.

$$E = \frac{4\pi\lambda^2\mu}{k^2} \quad \text{where} \quad \begin{array}{l} \lambda = \text{magnetostrictive constant} \\ \mu = \text{reversible permeability} \\ k = \text{electromechanical coupling coefficient} \end{array}$$

Composition

The first alloys developed for constant modulus purposes were binary iron-nickel compositions. Figure 1 shows that a zero temperature coefficient is obtained with alloys containing about 27% or 44% nickel, balance iron. These two alloys were found to be too sensitive to small changes in composition to be suitable for commercial production, a variation of 1% in nickel content shifting the coefficient of the 44% alloy about $50 \times 10^6/^\circ\text{F}$.

Addition of chromium to these iron-nickel alloys reduces sensitivity to composition but the resulting ternary alloys are still difficult to produce with the desired characteristics and require heavy cold reductions, seriously limiting sizes.

Addition of titanium to the iron-nickel-chromium

composition produces an alloy with a controllable thermoelastic coefficient, NI-SPAN-C alloy 902. This alloy is melted to the close compositional ranges shown in Table 1. The desired thermoelastic coefficient is then obtained by using cold work and the proper thermal treatment. Cold work produces internal strains, making the coefficient more negative. Thermal treatments, in the lower temperature ranges, relieve strain. They also cause complex ordering phenomena which make the coefficient more positive. Heating at temperatures above about 900°F causes the precipitation of an intermetallic compound of titanium and nickel, withdrawing nickel from the matrix and moving the coefficient further in the positive direction.

Table 1 - Limiting Chemical Composition, %

Nickel (plus Cobalt).....	41.0-43.5
Chromium.....	4.90-5.75
Titanium.....	2.20-2.75
Aluminum	0.30-0.80
Carbon.....	0.06 max.
Manganese	0.80 max.
Silicon	1.00 max.
Sulfur	0.04 max.
Phosphorus	0.04 max.
Iron	Balance*

*Reference to the 'balance' of a composition does not guarantee this is exclusively of the element mentioned but that it predominates and others are present only in minimal quantities.

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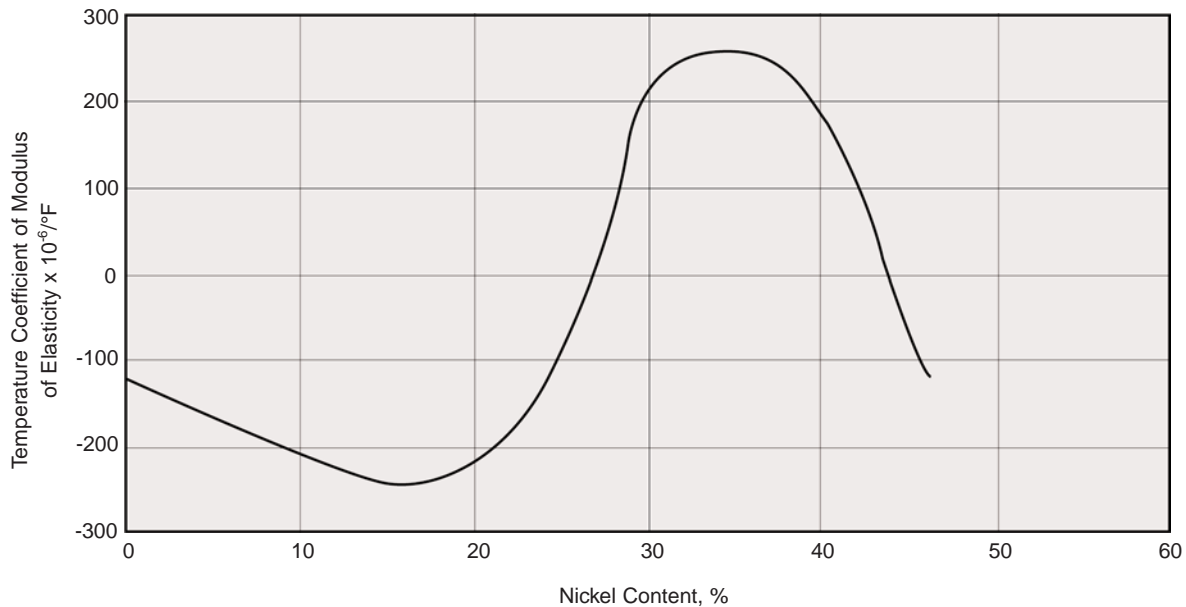


Figure 1. Effect of composition on the temperature coefficient of modulus of elasticity of iron-nickel alloys.

Thermoelastic Coefficient

The thermoelastic coefficient (TEC) of an alloy is the rate of change of its modulus of elasticity with change in temperature. It is usually expressed as parts per million per degree F (e.g. $5 \times 10^{-6}/^{\circ}\text{F}$).

The first measurements of the TEC of NI-SPAN-C alloy 902 were made using a torsion pendulum operating at about one cycle per second. These tests are the basis for much of the published data on the alloy. However, when these data were used to design high frequency devices they were found to produce incorrect results, and a confused situation arose.

Recent work has solved the problem of the conflicting results. It has been found that operating frequency has a marked effect on TEC. Tests run at Special Metals showed that the TEC of a given sample increased with increase in test frequency up to about 800 cps. Above 800 cps there was no frequency effect. Samples from the same heat of material

tested at about 1500 cps, and in another laboratory at 455,000 cps, gave identical results. Burnette at the National Bureau of Standards found that a heat treatment which produced zero TEC on a sample tested in a torsion pendulum at 1 cps developed a TEC of +40 on a sample tested in free-free vibration at 1000 cps. Figure 2 shows these data and a schematic indication of the frequency effect.

Because of the frequency effect, it is necessary to delineate two areas of application of NI-SPAN-C alloy 902, each requiring different processing to achieve best results. These are:

1. Low frequency devices. These include springs, Bourdon tubes, aneroid capsules, etc.
2. High frequency devices. Tuning forks, vibrating reeds, mechanical filters and similar instrumentation fall in this category.

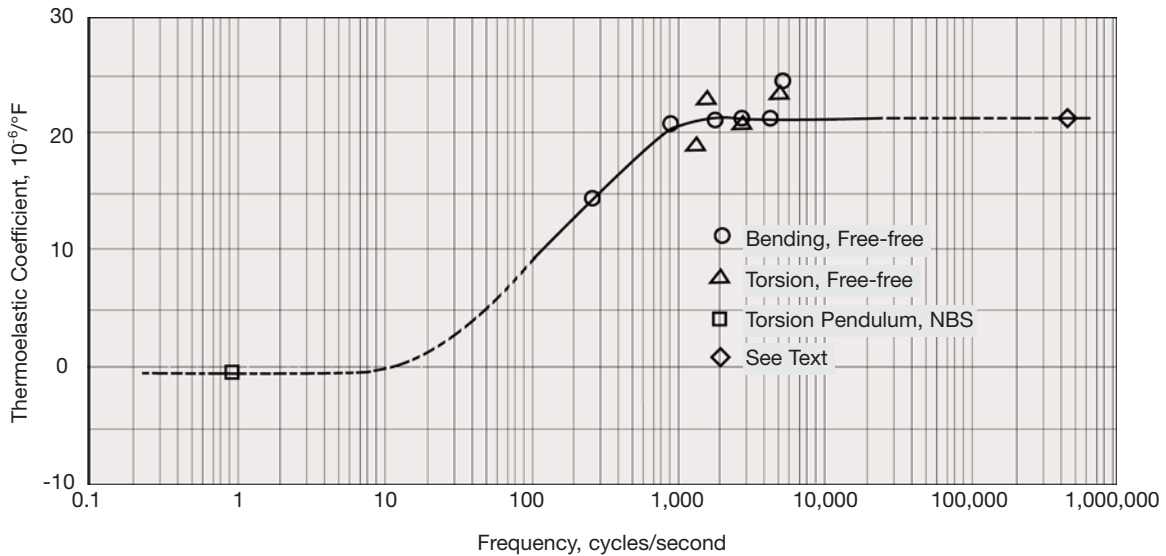


Figure 2. Effect of operating frequency on TEC. Free-free specimens heat treated 1200°F/5 hours, pendulum specimen 1285°F/3 hours. All specimens cold worked 50% before heat treatment.

Processing

The unavoidable differences in chemical composition between heats of the alloy lead to slight variations in elastic properties. These variations are considerably less than the overall accuracy of most devices in which the alloy is used, being on the order of ± 20 parts per million per degree F. For very precise applications, the effect of variation in composition may be adjusted by processing to obtain the desired TEC.

Low Frequency Devices

Low frequency devices usually require a low TEC, low mechanical hysteresis and low drift. This can be obtained by cold working about 35% and heat treating at 1100°-1200°F for 5 hours. All heats of the alloy, when given this treatment, will have a TEC suitable for most low frequency applications. Figure 3 shows that the lowest mechanical hysteresis is also obtained by this processing. Highest mechanical strength is another desirable result. (See Table 5.)

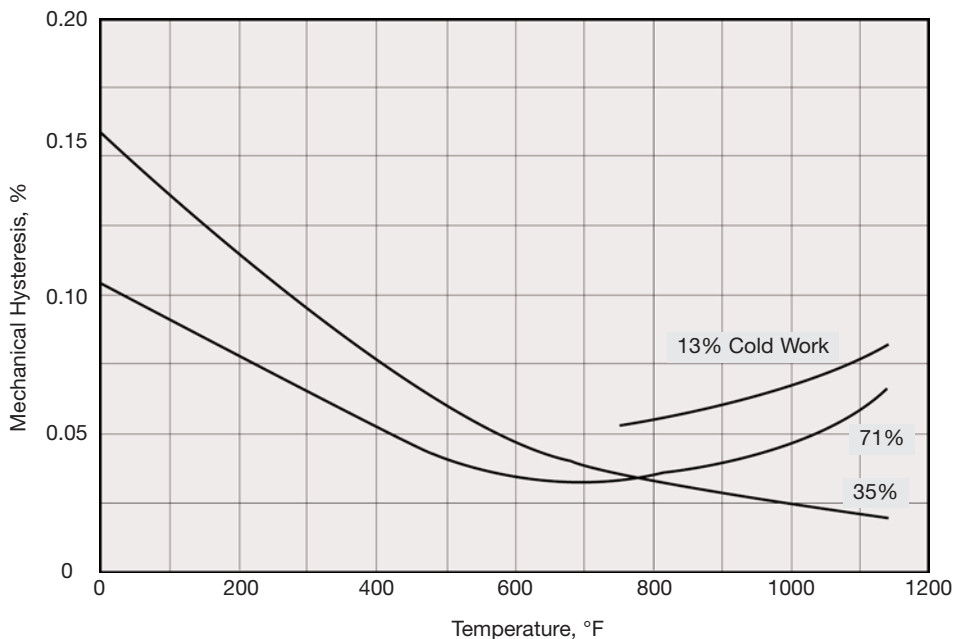


Figure 3. Effect of 5-hour heat treatment at temperature shown on torsional mechanical hysteresis.

NI-SPAN-C® alloy 902

Figure 3 also shows that the heat treating temperature must be lowered to 700° to 750°F for material with a high amount of cold work, if lowest mechanical hysteresis is desired. This heat treatment will result in a lower TEC.

High Frequency Devices

Very high precision equipment requires that each lot of material be pilot tested to determine the specific heat treatment necessary to produce the exact TEC required. A formula relating heat treatment temperature, composition and amount of cold work has been developed to assist in carrying out pilot tests. The formula, which applies only to high frequency applications, appears in the Appendix.

The effect of processing variables on the high frequency TEC of a typical heat is illustrated in Figures 4, 5 and 6. It will be noted that, for this heat, 50% cold work plus 5 hours at 860°F produced a zero TEC. Increasing the heat treating temperature to 1100°F produced a TEC 12 parts per million/°F higher.

Unless extreme precision is required, processing should be directed toward producing high strength, with its attendant fatigue resistance, and low mechanical hysteresis (30 to 50% cold work + 1100° to 1200°F/5 hours). The resultant TEC will be fairly close to zero.

Heat treatment temperature must exceed 600°F to insure stability of properties.

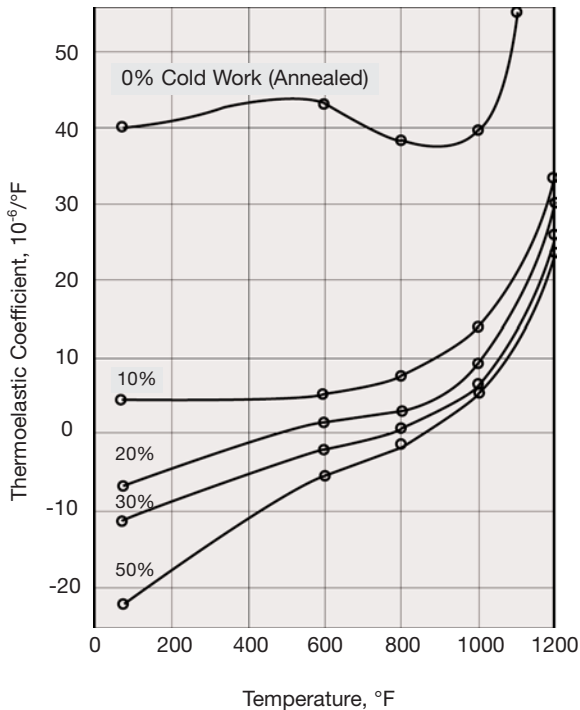


Figure 4. Effect of cold work and 5-hour heat treatment at temperature shown on thermoelastic coefficient.

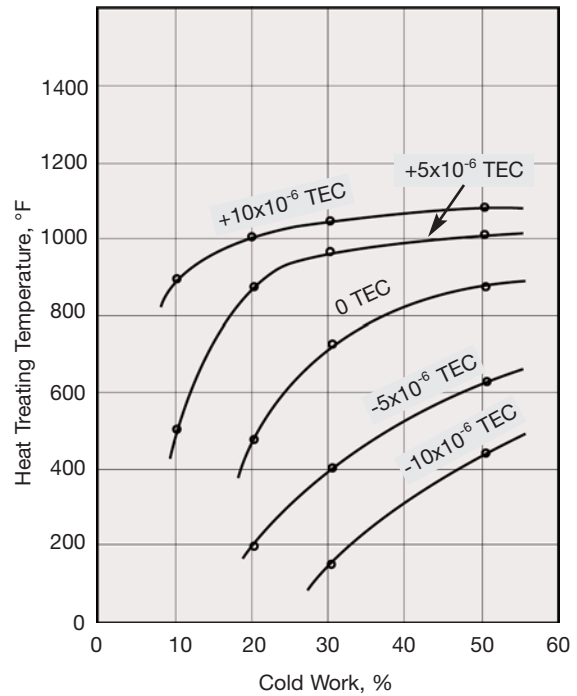


Figure 5. Cold work and 5-hour heat treatment required to produce various thermoelastic coefficient levels.

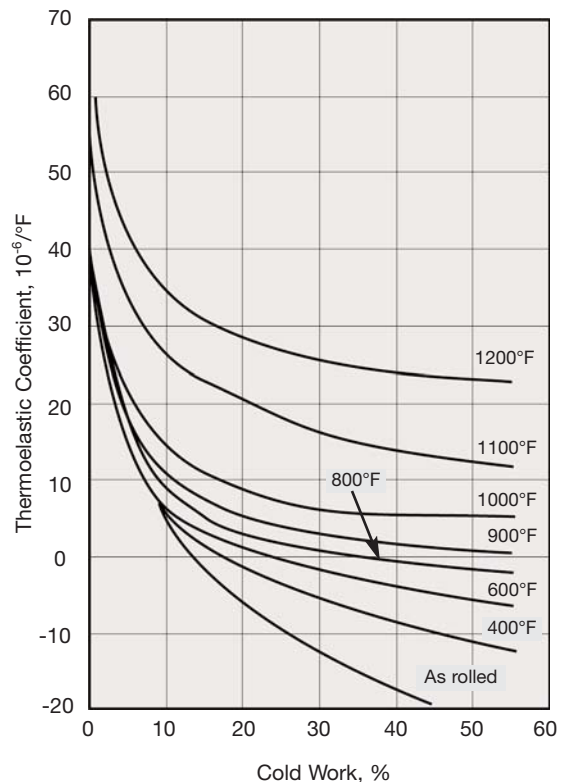


Figure 6. Effect of cold work and 5-hour heat treatment at temperature shown on TEC.

Properties

Physical Constants

Values for some of the basic physical constants are given in Table 2.

Thermal Conductivity

Thermal conductivity values for age hardened material (1260°F/6 hours) are listed in Table 3.

Table 3 - Thermal Conductivity

Temperature, °F	Thermal Conductivity BTU/sq. ft./hr/°F/in.
-238	52.7
-148	63.1
32	80.4
212	95.0
392	106.8
572	117.9
752	127.6
932	137.3
1004	141.4

Thermal Expansion

The average coefficients of expansion of one melt of the alloy with various heat treatments are shown in Table 4. It will be noted that there is no significant change in expansion characteristics due to heat treatment. Other tests have shown that the amount of cold work prior to heat treatment has only minor effect on expansion characteristics.

Figure 7 is a graphical representation of the effect of temperature on thermal expansion. A constant low rate of expansion is maintained over the useful working range. Then the curve breaks sharply upward until a comparatively high value is reached. The temperature at which the expansion rate begins to increase rapidly is known as the “inflection point” or “inflection temperature”. It can be determined graphically from the total expansion curve as shown in the figure. This behavior is typical of iron-nickel alloys of the low expansion type.

Table 2 - Physical Constants of NI-SPAN-C alloy 902

Density, lb/in ³	0.291
g/cm ³	8.05
Melting Range, °F	2650-2700
°C	1450-1480
Specific Heat, Btu/lb•°F	0.12
J/kg•°C	500
Curie Temperature, °F	380
°C	190
Electrical Resistivity, ohm•cmil/ft	611
μΩ•m	1.02

Table 4 - Thermal Expansion

Condition	Average Coefficient of Thermal Expansion, in./in./°F x 10 ⁶				
	-40 to 78°F	78 to 200°F	78 to 300°F	78 to 400°F	78 to 500°F
As hot rolled	4.5	4.5	4.6	5.3	6.0
1850°F/1 hr, WQ	4.2	4.3	4.4	4.8	5.5
1850°F/1 hr, WQ + 900°F/5 hrs, AC	4.3	4.2	4.3	4.9	5.6
1850°F/1 hr, WQ + 1000°F/5 hrs, AC	4.4	4.2	4.4	5.2	5.9
1850°F/1 hr, WQ + 1100°F/5 hrs, AC	3.9	4.2	4.6	5.5	6.0

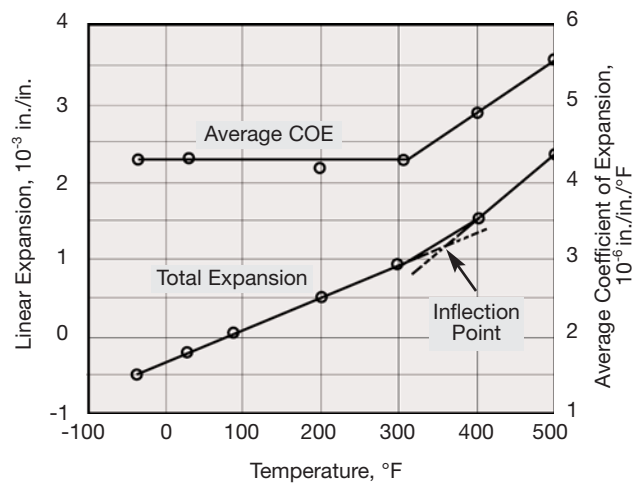


Figure 7. Thermal expansion characteristics. Hot rolled material, heat treated 1850°F/1 hour, WQ + 900°F/5 hours, AC.

Modulus of Elasticity

The tensile modulus of elasticity ranges between about 24 to 29 x 10³ ksi, depending on the processing. Torsional modulus range is about 9 to 10 x 10³ ksi.

Figure 8 shows the effect of cold work and thermal treatments on the room temperature tensile modulus. Cold work causes an increase in the modulus. Raising the temperature of heat treatment also causes an increase.

Figure 9 shows the effect of three heat treatments on the tensile modulus of cold rolled strip at different temperatures. The slope of these curves is the TEC. The figure shows how positive, zero and negative TEC values may be obtained from the same material by varying the thermal treatment. The sharp rise and fall of the curves above 250°F is due to the rapid change in magnetic properties above this temperature. The maximum points on the curves are approximate measures of the Curie temperatures of the materials tested.

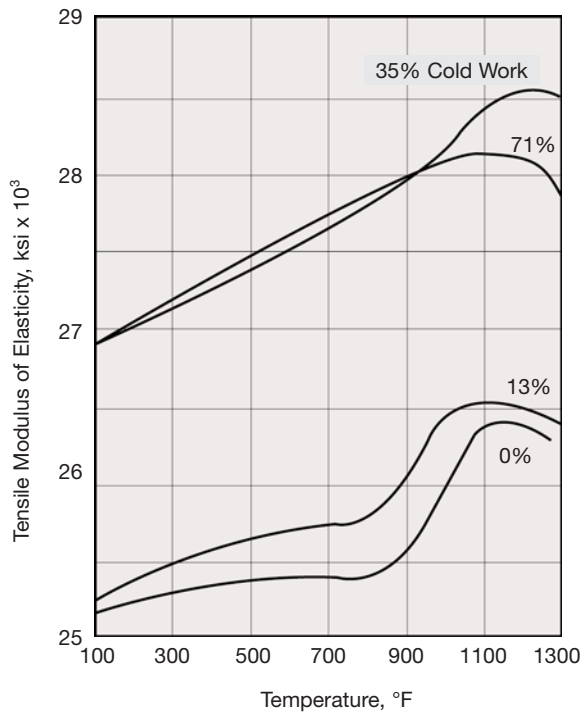


Figure 8. Effect of cold work and heating for 5 hours at temperature shown on the room temperature modulus of elasticity.

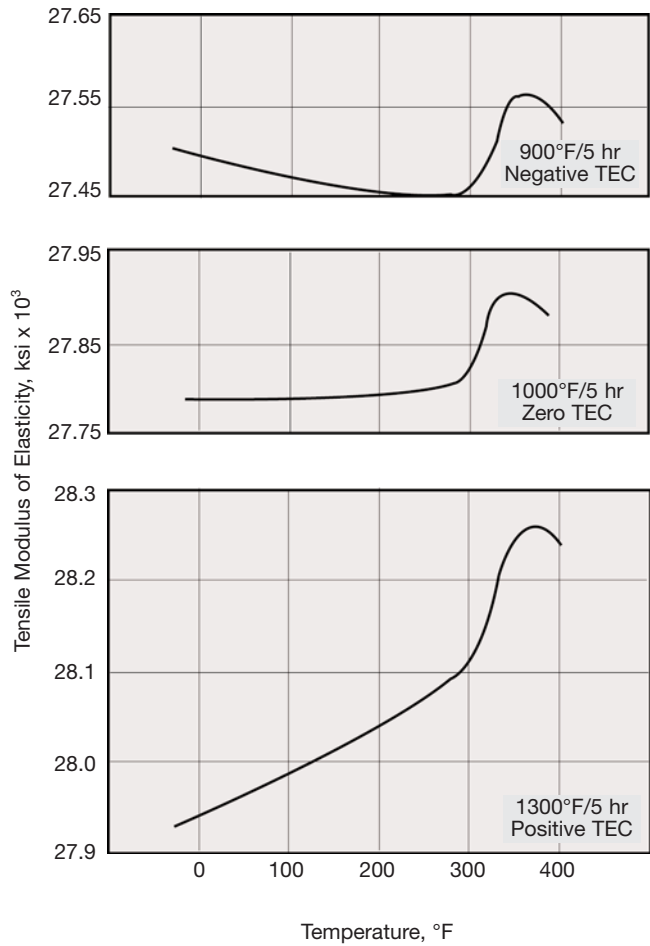


Figure 9. Effect of various heat treatments on the tensile modulus of elasticity at different temperatures. Material cold worked 50% prior to heat treatment.

Mechanical Properties

Room temperature tensile and hardness values are shown in Table 5 and Figure 10. The data show why it is desirable to heat treat at 1100° to 1300°F whenever possible. Room temperature fatigue strength appears in Table 6.

Although the alloy was designed for use above -50°F, it has been used successfully at lower temperatures and some cryogenic data have been published. An abstract of this work is given in Table 7.

The alloy exhibits low damping capacity (high Q). The Q of cold worked material has been reported to be about 8000, about 4 times greater than the values for annealed forms.

Table 5 - Tensile Strength and Hardness of Cold Rolled Strip^a

Condition	Tensile Strength, ksi	Yield Strength 0.2% offset, ksi	Elongation, %	Hardness Rockwell C
As rolled	131.0	126.0	6.5	26
500°F/5 hrs	139.0	136.0	7.0	29
900°F/5 hrs	140.5	135.0	11.0	30
1000°F/5 hrs	150.0	137.0	12.0	33
1100°F/5 hrs	178.5	165.0	9.5	37
1200°F/5 hrs	192.0	176.0	9.0	40
1300°F/5 hrs	193.0	173.0	8.5	40

^a 50% reduction.

Table 6 - Fatigue Strength

Form and Condition	Endurance Limit (10 ⁸ cycles), ksi
Bar, aged ^a	50
Sheet, cold rolled & aged ^a	50
Sheet, annealed ^b & aged ^a	40
Bar, aged ^a - torsion	18 (estimated)

^a1200°F/4 hours.

^b1850°F/1 hour.

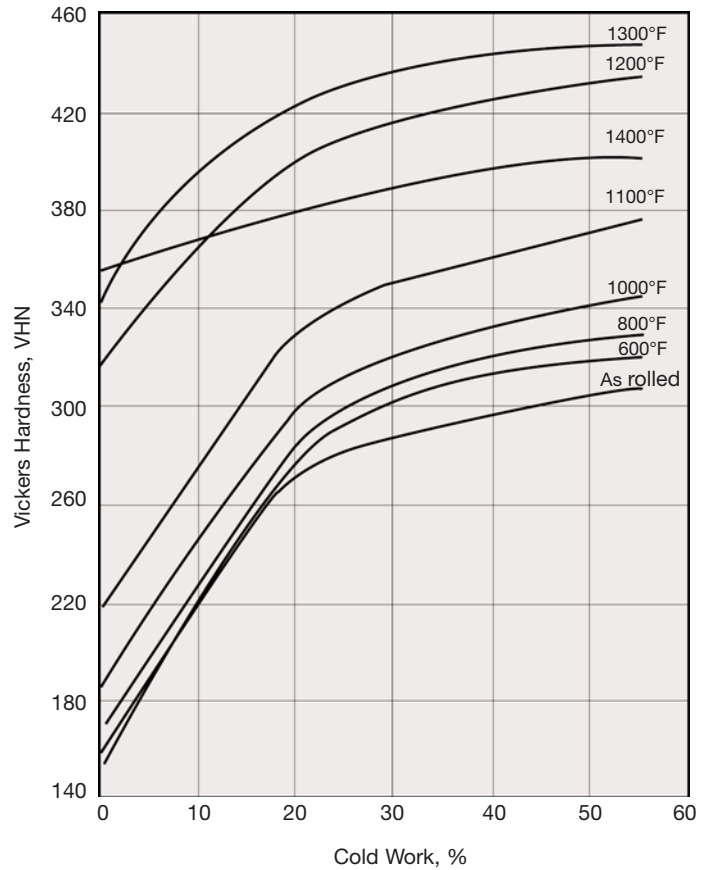


Figure 10. Effect of cold work and 5 hour heat treatment at temperature shown on room temperature hardness.

Table 7 - Properties at Low Temperatures

Property	Condition	Temperature, °F		
		70	-200	-423
Tensile Strength, ksi	Hot rolled, aged	175	205	245
Yield Strength, ksi	Hot rolled, aged	110	125	145
Elongation, %	Hot rolled, aged	25	29	30
Reduction of Area, %	Hot rolled, aged	50	48	44
Modulus of Rigidity (G), 10 ³ ksi	Hot rolled, aged	10.19	10.10	10.06
Modulus of Elasticity (E), 10 ³ ksi	Hot rolled, aged	25.1	24.9	24.7
Fatigue Strength (10 ⁶ cycles), ksi	Cold rolled, aged	80	107	122
Impact Strength, Charpy U, ft lbs	Hot rolled, aged	18	17.5	17

Mechanical Hysteresis

When a spring is loaded and then unloaded, the load-deflection curves do not coincide, even though the elastic limit of the material is not exceeded. This departure from linear elastic behavior, termed mechanical hysteresis, is illustrated in Figure 11. It is expressed quantitatively by the expression:

$$\% \text{ hysteresis} = \frac{100 \times \text{maximum width of hysteresis loop (AB)}}{\text{maximum deflection (OC)}}$$

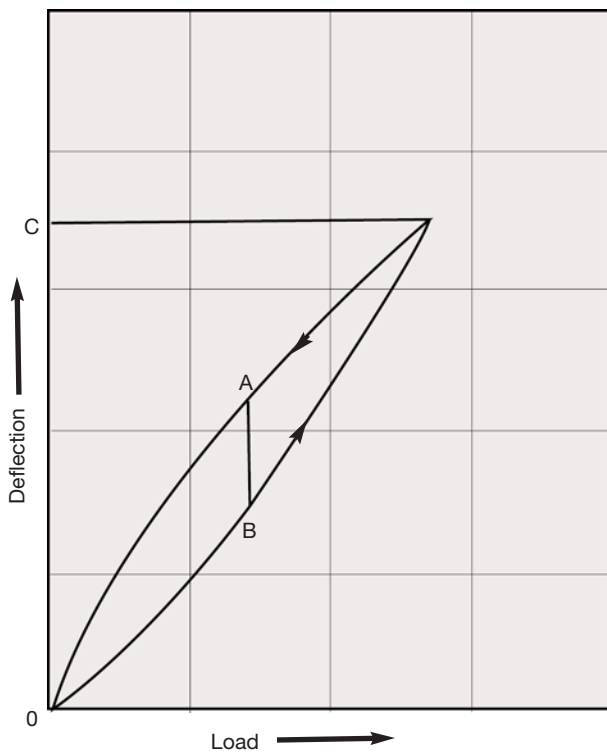


Figure 11. Schematic Load-Deflection curve.

Mechanical hysteresis values as low as 0.02% have been obtained for NI-SPAN-C alloy 902 springs by controlled cold work and heat treatment. The effect of these variables is shown in Figure 3. It will be noted that increase in amount of cold work decreases hysteresis and that increase in heat treatment temperature has a generally similar effect. Minimum hysteresis and maximum strength for the alloy are obtained by cold working 30 to 50% and heat treating at about 1100° to 1200°F.

The value of mechanical hysteresis depends on maximum loading stress, increasing with increase in loading stress. The data shown in Figure 3 were obtained for a maximum torsional loading stress of 25 ksi, which is typical of normal practice, when fatigue is not a factor.

Magnetostrictive Properties

The alloy has low magnetostrictive properties. However, they are quite adequate for some applications, such as delay lines.

Magnetic Properties

The saturation magnetization at room temperature is approximately 5000 gauss. Permeability is affected by cold work and heat treatment but no extensive investigation has been made to establish values. Figure 12 shows a normal magnetization curve for cold rolled and aged material. The effect of magnetic field intensity on the TEC is illustrated in Figure 13.

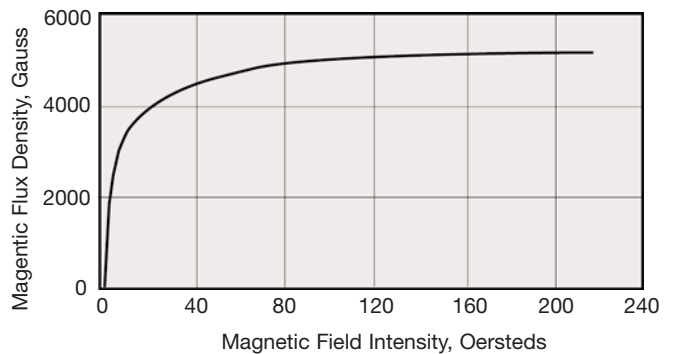


Figure 12. Normal magnetization curve. Material cold rolled 40% and heat treated 1000°F/5 hours prior to testing.

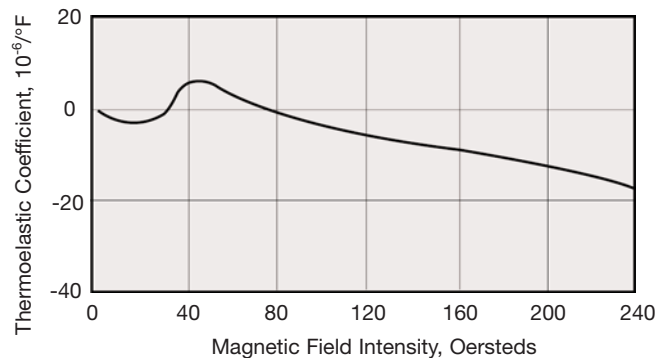


Figure 13. Effect of magnetic field intensity on thermoelastic coefficient. Material cold rolled 40% and heat treated 1000°F/5 hours prior to testing.

As in all ferromagnetic alloys, the modulus of elasticity is affected by magnetization. As magnetic intensity is increased, modulus decreases until the knee of the magnetization curve is reached. Above this point the trend reverses and modulus increases until magnetic saturation is reached. At higher temperatures these effects occur at lower field strengths. See Figure 14.

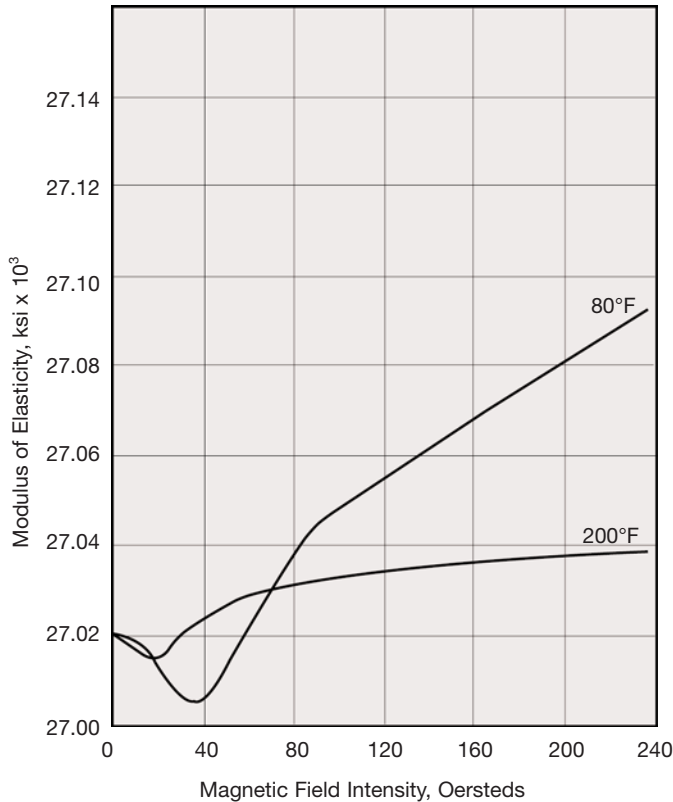


Figure 14. Effect of magnetic field intensity on modulus of elasticity at two temperatures. Material cold rolled 40% and heat treated 1000°F/5 hours prior to testing.

Working Instructions

Heat Treating

In order to maintain bright surfaces during final heat treatment the process must be carried out in a high vacuum or in a very pure hydrogen atmosphere. Experience has shown that an absolute pressure of 0.1 micron or less is required for bright work in vacuum furnaces. Pressures around 1.0 micron may produce a faint straw color. Ultrapure hydrogen, such as that produced by palladium diffusion cells, will also maintain brightness.

Annealing

The alloy, as usually supplied by the manufacturer to the user, is carefully processed and ready for heat treatment after fabrication. If, for any reason, it is desired to soften the material by annealing, the following instructions should be carefully observed:

Heat at 1850°F in a reducing atmosphere free from sulfur and quench rapidly. Delay in quenching will result in partial hardening of the alloy and subsequent adjustment of the TEC by cold work and heat treatment will be impaired. Annealing time is governed by the dimensions of the work.

Pickling

Thin oxide films formed by heating the alloy in atmospheres less pure than ultrapure hydrogen or in vacuums lower than those cited can be removed by mechanical abrasion or by pickling. Pickling the final product will result in a decrease in fatigue strength; therefore abrasive blasting or mechanical polishing should be considered for components which require best fatigue resistance.

Pickling can be accomplished by dipping the material in Formula 4 for about 15 minutes, rinsing, dipping into Formula 9, rinsing and drying. The electrolytic pickle, Formula 15, may also be used. It is particularly convenient for continuous processing.

Formula 4

Water	1 gal	1000 ml
Sulfuric acid (66° Be)	¾ pt	95 ml
Sodium nitrate (crude)	½ lb	55 gm
Common salt	1 lb	110 gm
Temperature	180°-190°F	82°-88°C

Formula 9

Water	1 gal	1000 ml
Nitric acid	1 qt	250 ml
Temperature	140°-160°F	60°-71°C

Formula 15

Water	1 gal	1000 ml
Sulfuric acid (66° Be)	¾ pt	95 ml
Sodium fluoride	3 oz	25 gm
Temperature	Room	
Current density	50 to 100 amps.sq.ft.	

Joining

The alloy can be joined by welding, brazing, or soldering if the surfaces to be joined have been thoroughly cleaned. The material should be brazed in the heat treated condition in order to prevent cracking. Autogeneous inert gas fusion welding has been employed successfully.

In any joining operation the effect of joining temperature and time must be considered in relation to its effect on the thermoelastic characteristics of the device.

Corrosion Resistance

NI-SPAN-C alloy 902 is not “stainless”, as it will acquire an adherent red-brown oxide film when exposed to outdoor environments. Tests in industrial and marine atmospheres have shown corrosion rates of less than 0.001 inch per year.

Available Products and Specifications

NI-SPAN-C nickel-iron-chromium alloy 902 is designated UNS N09902. The alloy is produced in the form of round bar and strip. Wire, thin gauge strip, round tube, and special Bourdon tubular shapes are available from convertors.

There are three AMS specifications covering the alloy:

- SAE AMS 5221 - Strip, solution annealed
- SAE AMS 5223 - Strip, 10% cold rolled
- SAE AMS 5225 - Strip, 50% cold rolled

Applications

The alloy is used in many types of precision apparatus where elastic members are subject to temperature fluctuations. Resonant vibrating systems such as electro-mechanical filters, tuning forks and vibrating reeds are typical examples of devices where a constant frequency is desired. A zero TEC material may be used for the vibrating member or a slightly positive or negative TEC may be chosen to compensate for thermal drift due to other components in the device.

Another major application is springs. Here the constant TEC property makes deflection independent of temperature. Examples are Bourdon tubes, aneroid capsules, geophysical instruments, hairsprings for timing devices, diaphragms, and springs for weighing instruments.

Appendix

Prediction of Heat Treatment Temperature for Desired Thermoelastic Coefficient (NI-SPAN-C alloy 902)

A formula has been developed for predicting the heat treatment required to produce the desired TEC for a specific lot of material. The formula was derived by multiple graphical correlation analysis of experimental data obtained at a frequency of about 1500 cycles/second, and will give a fairly close approximation within the limits shown. Pilot tests will still be required for high precision applications but use of the following equation will simplify testing.

$$TEC = -12.05 Ni - 16.45 Cr + 10.00 Ti + A + B + 628.68$$

where:

Ni = Nickel, % by weight	<u>Limits</u> 41.0 to 43.5
Cr = Chromium, % by weight	4.90 to 5.75
Ti = Titanium, % by weight	2.20 to 2.75
A = Effect of heat treating temperature	600° to 1300°F
B = Effect of cold work	0 to 100%

Table A lists the values of factor A for various temperatures and Table B gives values of factor B for various amounts of cold work.

Normally all variables except heat treating temperature are known. Solve the equation for factor A, then determine the predicted temperature by inspection of Table A. An alternate procedure is to calculate the various values of TEC which would result from different temperatures, plot the results, and choose the desired temperature from the resulting graph. This temperature is used for the first pilot test. Final adjustment of temperature is based on test results.

Table A
Values for Factor A in TEC Formula

Temperature, °F	Factor A
600	-81.3
700	-79.6
800	-78.0
850	-76.5
900	-75.1
950	-73.5
1000	-71.2
1050	-68.8
1100	-65.3
1150	-61.7
1200	-55.7
1250	-53.5
1300	-51.7

Table B
Values for Factor B in TEC Formula

Cold Work, %	Factor B
0	29.7
5	21.7
10	20.3
15	19.4
20	18.7
25	18.2
30	17.8
35	17.4
40	17.1
45	16.8
50	16.5

Determination of Thermoelastic Coefficient

The apparatus used in the laboratory of Special Metals Corporation to determine TEC is based on that described by Roberts and Northcliffe. A carefully measured specimen is suspended by two strings. Each string terminates in a high output crystal phonograph pickup. One pickup is driven by a variable frequency audio oscillator while the other accepts vibrations from the specimen through its string and feeds its output into a sensitive AC vacuum tube voltmeter. In operation the sample is hung in a small chamber provided with accurate temperature measuring and controlling equipment. After the system has reached thermal equilibrium at the chosen temperature, the operator sends an alternating current through the crystal driver and observes the output signal magnitude on the voltmeter. The signal frequency is varied to obtain maximum output voltage, which indicates resonance. The resonant frequency is then accurately determined with an electronic counter. Resonant frequency for a rectangular sample is related to its dimensions and the tensile modulus of elasticity by the equation:

$$f = \frac{K_t}{L^2} \left[\frac{E}{d} \right]^{1/2}$$

where f = resonant frequency
 K = a constant which depends on mode of vibration
 t = thickness of strip, in.
 L = length of strip, in.
 d = density of material, lb/cu. in.
 E = modulus of elasticity in tension (Young's Modulus), psi

The thermoelastic coefficient can be derived directly from the plotted data using the expression:

$$TEC = \frac{2}{f} \frac{df}{dt}$$

where f = resonant frequency
 $\frac{df}{dt}$ = slope of the plotted frequency vs. temperature curve

All tests conducted in the Special Metals laboratory employ a specimen 0.125 x 0.250 x 4.00 inches in dimension, vibrated in its fundamental mode of bending. This requires a test frequency of about 1500 cycles/second.

Note: The method of testing described actually measures the change in stiffness of a given specimen with change in temperature. Change in stiffness has two components, change in elastic modulus and thermal expansion effects. It is necessary to correct for thermal expansion if absolute values of TEC are required. In practice, change in stiffness is the property which governs actual performance of a device. For this reason, thermal expansion effects are usually ignored. In this publication thermoelastic coefficient (TEC) is defined to be the change in elastic modulus uncorrected for thermal expansion effects.

Velocity of Sound

The theoretical velocity of an extension wave in a wire, such as in a delay line, is given by:

$$V = C \left[\frac{E}{p} \right]^{1/2}$$

where V = velocity of sound, inches/second
 E = modulus of elasticity in tension, psi
 p = density, pounds/cu. in.
 C = 19.66

For the range of E obtained in NI-SPAN-C alloy 902 (24 to 29 x 10³ ksi) the formula predicts a velocity range of 178,000 to 196,000 inches/second. Two actual test results showed values of 187,500 to 188,000 inches/second confirming the validity of the equation.



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